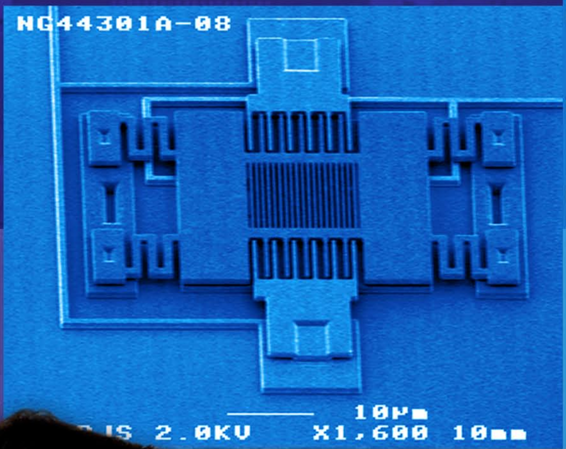
LABORATORY DIRECTED RESEARCH AND DEVELOPMENT

2006 LDRD ANNUAL REPORT

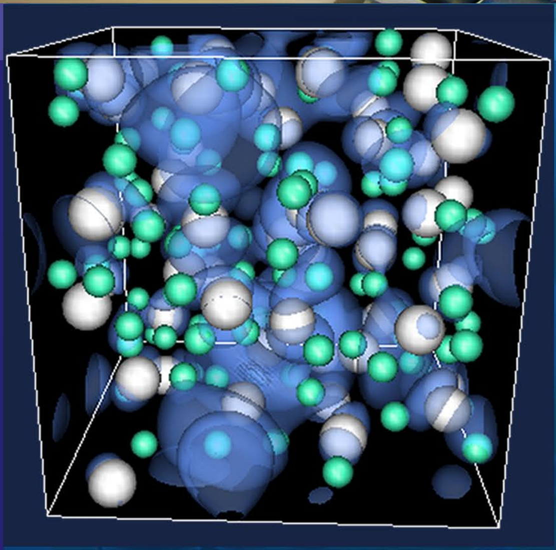


Science, Technology, and Engineering

Mission Technologies

Corporate Investments

Grand Challenges



NNSA National Nuclear Security Administration

SAND2007-1774

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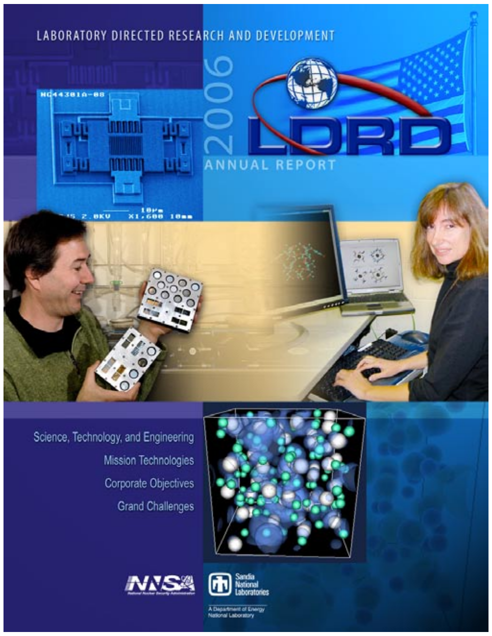

Cover photos (middle right going clockwise):

(1) Sandia researcher Susan Rempe uses Density Functional Theory (DFT) computer codes to identify mechanisms of ion permeation in potassium channels.

(2) Structure of conducting water together with electron density from a partially occupied band.

(3) Sandia researcher Mat Celina displays the piezoelectric polymer films that will be tested on an upcoming Materials International Space Station Experiment.

(4) Ultra-sensitive nanophotonic motion sensor.

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Abstract

This report summarizes progress from the Laboratory Directed Research and Development (LDRD) program during fiscal year 2006. In addition to a programmatic and financial overview, the report includes progress reports from 430 individual R&D projects in 17 categories.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94-AL85000.

SAND 2007-1774
March 2007

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Sandia National Laboratories' FY 2006 Laboratory Directed Research and Development (LDRD) Program: Program Improvements!

Richard H. Stulen, Vice President of Science, Technology, and Engineering and Chief Technology Officer

Sandia National Laboratories provides world-class science, technology, and engineering in support of the national security missions for the National Nuclear Security Administration (NNSA), the Department of Energy (DOE), and Other Federal Agencies (OFA).

It is essential that Sandia maintain its leadership role in science and technology (S&T) to address emerging threats to our national security and economic prosperity. Sandia's value to the nation is linked directly to our goal to become the 'Lab of Excellence' for S&T with renewed emphasis on technical innovation.

"Are we - and are we perceived to be - places of unique excellence for science and technology?"
(Sandia President and Director Tom Hunter)

The Laboratory Directed Research and Development (LDRD) program is the cornerstone of the S&T vitality of the Labs; it's what enables Sandia to distinguish itself as a world leader in S&T fields. The program nurtures an environment of vibrant scientific curiosity, creativity, and innovation, and its investment in leading-edge R&D provides great benefit to current and future sponsors. These investments are a precious resource to the Labs, and we must constantly strive to eliminate waste while improving quality and overall impact. Because LDRD success is essential to achieving the Labs' strategic goals, the demand for excellence in the management, execution, and application of R&D is very high.

FY 2006 was a transition year that ushered in many improvements to the program regarding strategic intent, project selection, and managing outcomes.

One key improvement was instituting Discover, Create, and Prove (DCP) categories as a way to clearly describe the purpose of the proposed R&D.

Winners of FY 2006 LDRD Awards for Excellence: T. Tai, U. Krishnamoorthy, M. Desjarlais, R. Stulen (CTO) , H. Masood (for A. Singh), G. Sloan, G. Grest

- *Discover* is the creation of new understanding or new knowledge (use-inspired research).
- *Create* is the innovative application or combination of new or existing knowledge in a unique way to create a novel solution to a problem, or to provide a revolutionary advance in a critical ST&E area (use-driven R&D).
- *Prove* is the proof of a prospective innovation or concept in a real-world environment of interest that reduces the remaining unknowns and uncertainties (use-driven development).

Setting DCP funding targets for the whole program provided R&D balance across the LDRD portfolio and identified reasonable expectations of individual project success.

Another major strategic effort was to consolidate Investment Areas (IA) for FY 2007 by about a third in order to better align IA strategic goals with the new corporate goals. We also explored new opportunities to partner with academia in order to promote and enhance our technological innovations in the fields of nanoscience, modeling and simulation, and engineering.

We formed the Senior Steering Committee (SSC), comprised of senior managers from each IA, to provide greater integration and communication across the LDRD program. The SSC managed the FY 2006 LDRD portfolio of projects and was instrumental in the IA reorganization and the project selection process for FY 2007.

There was increased down selection of the ideas to achieve a reduced 2:1 ratio of proposals for every project selected. While difficult up front, this new process saved significant time and effort on the part of staff and management since fewer proposals were written and reviewed. Technical review comments were provided to staff prior to oral proposal defenses, which enabled greater transparency and effectiveness in the decision process. The SSC and I reviewed the entire LDRD portfolio to ensure a consistency and quality across the program.

Lastly, to better leverage the outcomes of the R&D, we began collecting and evaluating new metrics to communicate project outcomes. Out of this effort arose the first-ever LDRD Day and the opportunity to share results, promote networking, recognize accomplishments, and distribute LDRD Awards for Excellence. Many of the projects were showcased at the event specifically for the significant impact each had made in such important areas as high-performance computing, biotechnology, and remote sensing:

- Breakthrough density functional theory for high energy density physics applications
- Ultrasensitive nanophotonic motion detection sensors using nano-g accelerometers
- Enhanced synthetic aperture radar applications using novel radar software architectures and algorithms
- Identification of protein functions via high-throughput proteomics technologies
- Analysis and understanding of the vulnerabilities in complex systems and networks
- Multiscale modeling and prediction of wetting behavior in multicomponent systems

All these changes were critical steps toward achieving the high-level goals for LDRD, including a renewed emphasis on differentiating science, a less risk-averse R&D portfolio, and innovation in alignment with Sandia's strategic missions. We are making improvements, but we cannot settle for anything less than the best from this precious resource…Sandia's future depends upon it.

"...exceptional service in the national interest."

"Science and technology are the heart of the United States industrial competitiveness, national security, energy resources, environmental quality, and leadership in fundamental and applied science."

"To all those who have contributed so generously of their time and talent, thanks and congratulations for a job well done."

An Overview of Sandia's FY 2006 Laboratory Directed Research and Development (LDRD) Program

K. Ortiz

The LDRD program is authorized by Congress to maintain the vitality of the national laboratories in national security-related scientific disciplines. The program is directed by Sandia within the constraints of DOE Order 413.2B, *Laboratory Directed Research and Development*. LDRD is differentiated from R&D directed by DOE, NNSA, and other sponsors in several ways:

LDRD is employee-suggested R&D. LDRD is a "bottom-up" approach, initiated by the employees who are the closest to current R&D and the best able to identify promising new directions.

LDRD fills technical needs not funded by ordinary programs. LDRD projects could have high technical risk that may be intolerable to program sponsors or they could focus on important science and technology that are not of interest to program sponsors.

LDRD anticipates future mission needs. LDRD projects could lay the groundwork for future program sponsored work, where the prospective sponsor may not yet be ready to fund the need or the prospective sponsor may not yet exist.

LDRD is flexible. Opportunistic R&D may be rapidly funded and a project's scope may be changed so that the R&D paths of greatest value may be pursued.

LDRD generates intellectual property. LDRD projects create intellectual property that may be applied at Sandia's discretion to the benefit of all customers of the Laboratories.

These unique features are critical to maintaining the vitality of the Labs where ordinary, program funded work by itself would not. Without LDRD, Sandia would fall behind scientifically and technically, would not be prepared for future work, and would not be an attractive place for the best and the brightest to work. Thus, LDRD is Sandia's most precious R&D funding resource.

We constantly strive to increase the value of the LDRD program to Sandia and to the Nation. This past year, FY 2006, was a year of important changes to the program. In the course of this overview, we will mention a few significant and externally visible changes.

Discover! Create! Prove!

We are most excited about the creation of three new categories of projects that cut across the customary Investment Areas (IAs) to facilitate investment portfolio balancing. LDRD projects span the entire "fuzzy front end" of innovation, from discovery through concept demonstration. The LDRD portfolio seeks to balance, on one hand, the creation of new knowledge and, on the other hand, the application of existing knowledge to solve practical problems; the assurance of the long-term future of the Labs versus meeting short-term needs; and the pursuit of high-risk, out-of-the-box ideas versus the reduction of risk to potential follow-on projects.

Beginning in the FY 2006 program, LDRD projects are placed into three categories, Discover, Create, and Prove.

Discover: The creation of new understanding or new knowledge (use-inspired research). Projects that "discover" focus on observing and theorizing.

Create: The innovative application or combination of new or existing knowledge in a unique way to create a novel solution to a problem or to provide a revolutionary advance in a critical ST&E area (use-driven research and development). Projects that "create" focus on invention and insight.

Prove: The proof of a prospective innovation or concept in a real-world environment of interest that reduces the remaining unknowns and uncertainties (use-driven development). Projects that "prove" focus on concept demonstration in environments (actual or simulated) relevant to a prospective application.

We created an award to recognize work that exemplifies Discover, Create, and Prove. Nominations for the LDRD Awards for Excellence were evaluated for their embodiment of "National Laboratory Challenge, Risk and Creativity" and of "National Laboratory Relevance and Impact." This inaugural year, six principal investigators (PIs) were presented with awards, one for work in the Discover category, four in Create, and one in Prove. The winners are shown in the photograph in Rick Stulen's Introduction.

Discover:
Gary Grest established a multiscale competence for characterizing and predicting the wetting behavior of polymer blends by combining atomistic and continuum level modeling with experimental studies and validation. ("Elucidating the Mysteries of Wetting," project 52528, FY 2003-2005)

Create:
Michael Desjarlais provided a breakthrough capability in condensed matter and high energy density sciences by efficiently implementing finite-temperature, exact-exchange density functional theory in Sandia's Socorro code. ("Beyond the Local Density Approximation: Improving Density Functional Theory for High Energy Density Physics Applications," project 79878, FY 2004-2006)

Uma Krishnamoorthy conducted groundbreaking work on the design and development of nano-g accelerometers using nanophotonic motion detection sensors. ("Nano-g Accelerometers Using Nanophotonic Motion Detection System," project 67023, FY 2004-2006)

Anup Singh pioneered and established the use of high-throughput proteomics technologies at Sandia to study protein-protein interactions and elucidation of protein function. ("High Throughput Identification of Molecular Machines Involved in Membrane Signaling and Toxin Pathways," project 52536, FY 2003-2005)

Tan Thai contributed significantly to national security through the innovative melding of disparate ideas to create novel, classified solutions. ("Infrastructural Development for Flexible Network of Devices," project 67087, FY 2004-2006)

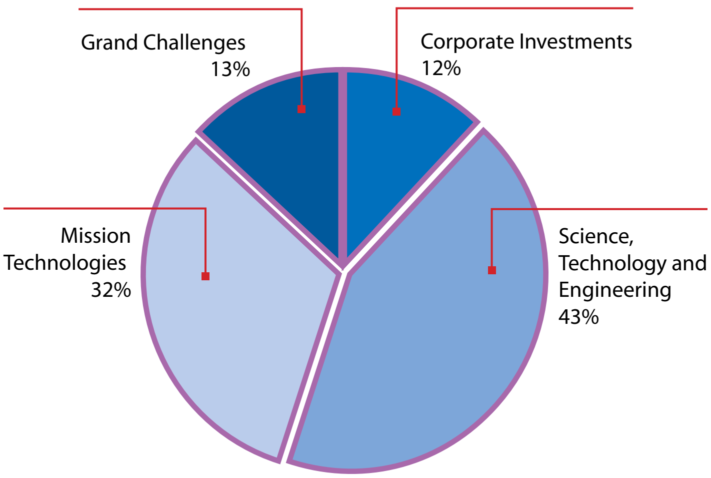
Prove:
George Sloan supported the advancement of radar for intelligence, surveillance and reconnaissance (ISR) through innovation excellence in novel radar software architectures development, ingenuity in complex field programmable gate array (FPGA) algorithm implementation approaches, and development of other enabling capabilities. ("Novel Processing, Affordable Motion Compensation, and Mode Multiplexing for Miniaturized Synthetic Aperture Radar," project 67035, FY 2004-2006)

In recognition of the fact that a project's impact is often not known for several years after its completion, projects were eligible for this award if they were completed in any one of the last three fiscal years (i.e., FY 2004, 2005 or 2006). Note that two of the awards went to projects that finished in FY 2005 and four in FY 2006.

We also launched an annual event, LDRD Day, to exhibit noteworthy projects that ended in FY 2006. Approximately 200 Sandians attended the first LDRD Day on November 3, 2006, where nine oral presentations and twenty-three posters were featured. In addition to showcasing these projects, the event provided an opportunity for PIs to network and communicate with each other outside of normal work about LDRD. As a result, many new ideas for LDRD projects and potential applications for the LDRD on display were explored.

Creating the FY 2006 Portfolio
The FY 2006 program began to take shape in January 2005, when Sandia's Executive Office approved the structure of the FY 2006 program. The approved structure reflects the corporate strategic management units at the time.

As in years past, the FY 2006 portfolio includes four Program Areas (PAs): Science, Technology, and Engineering (ST&E), Mission Technologies (MT), Grand Challenges (GC), and Corporate Investments (CI).



Investments in ST&E could support any or all the missions of the Laboratories. These LDRD projects typically address fundamental problems and enable solutions across multiple mission areas. For example, “Substructured Multibody Molecular Dynamics” (project 67017) advances capabilities to simulate the dynamics of biological molecules and nanoparticles through substructuring techniques, making possible simulations of molecular phenomena that would otherwise be computationally prohibitive. ST&E is divided into the following Investment Areas (IAs):

Advanced Components and Certification Engineering
Advanced Manufacturing
Biotechnology
Chemical and Earth Sciences
Computational and Information Sciences
Electronics and Photonics
Engineering Sciences
Materials Science and Technology
Pulsed Power/High Energy Density Sciences
S&T Strategic Objectives

Investments in MT typically support the anticipated needs of Sandia’s missions more directly. These projects are typically nearer-term and have more obvious relevance to Laboratory missions. For example, “Water-Splitting Nanodevices for Solar Hydrogen Production” (project 93563) has clear relevance to the energy R&D mission. It investigates the creation and properties of self-assembled porphyrin nanostructures that use sunlight to directly split water into hydrogen and oxygen, potentially a fundamental breakthrough in establishing a hydrogen economy. MT is divided into the following IAs:

Energy and Infrastructure Assurance
Homeland Security
Military Technologies and Applications
Nonproliferation and Assessments

Note that, while the MT Investment Areas correspond to the Sandia Strategic Management Units, LDRD projects benefit multiple missions and a particular beneficiary is not assignable. Note also that we expect the ST&E portfolio to be slightly weighted toward Discover and MT toward Prove. However, we have set investment targets for all three categories in each PA. The majority of projects in both PAs will be in Create.

The Grand Challenges are large (greater than $1M), multidisciplinary projects that address the difficult and complex R&D issues that are vital to Sandia’s core purposes. They could be either ST&E-like or MT-like or both in character. For example, “Terahertz Microelectronic Transceiver (TμT) System” (project 95214) is working to integrate quantum cascade lasers and other devices into a chip-level integrated transceiver for electromagnetic radiation in the terahertz frequencies, i.e., between microwave radio and far infrared light. Key breakthroughs in science and technology are necessary for the project to be successful, but numerous practical applications would become immediately feasible. This project demonstrates beautifully how, due to their large size, GC projects can marshal the resources of a national laboratory in ways that normal projects cannot.

Investments in the relatively small Corporate Investments area support a variety of Laboratory needs that are not met through the other investment areas. These needs include strengthening our ties with universities and funding ideas outside the scope of the other investment areas. For example, "Modeling and Simulation of Spectra Expected from Radiation Sensors Made from Arrays of MEMS Scale Capillaries" (project 103004) is a Seniors' Council project that is exploring a high-risk concept for detecting ionizing radiation using micro-scale tubes to hold the detecting gas at very high pressures, near liquid density, for example, 150 micron diameter tubes at 50,000 psi. The project is predicting the performance of such tubes and validating the computational models with experimental prototypes. If successful, pager-sized gamma-ray or neutron spectrometers could eventually be made with broad impact to NNSA, DOE, DHS, and others. CI is divided into the following IAs:

Advanced Concepts
Seniors' Council
University Collaborations

For ST&E, MT, and GC, the normal project selection process runs for three months each year, from March through May. Sandia employs a two-phase selection process to efficiently assure technical quality and mission relevance.

The first phase of selection is the Idea phase. The Investment Areas call for ideas for R&D projects. The Calls describe at a high level the strategic interests of the IAs including scientific and technical areas and potential applications and problems to be solved. The calls are open and stimulate many ideas. For FY 2006, 1233 one-page ideas were submitted for consideration. The brevity of the idea description minimizes the time spent by PIs and reviewers on unsuccessful proposals.

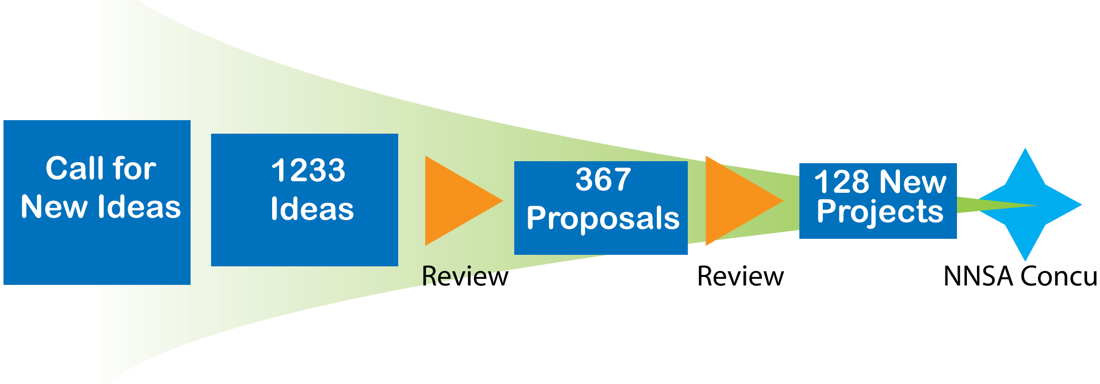
The second phase of selection is the Proposal phase. The IAs cull through the ideas and request full proposals for the most promising. For FY 2006, 367 six-page proposals were written. At least two reviewers, one for technical merit and one for programmatic merit review all proposals. The technical review ensures technical quality by evaluating a proposal's scientific and technical soundness, creativity and innovation, and project plan. The programmatic review ensures alignment with the strategic directions of the IAs and relevance to Sandia's missions.

Through this normal process, 128 new projects (the top ten percent of submitted ideas) were selected to begin in FY 2006. These projects joined 99 projects continuing into their second year and 64 into their third year. All continuing projects were reviewed in parallel to the normal process for technical progress and continuing programmatic alignment.

Before any LDRD project may begin, the NNSA performs an independent validation of the relevance of each LDRD project to the missions of NNSA and must provide its concurrence. Sandia submits to NNSA summaries of all the projects it wishes to start with the new fiscal year by the first of September each year.

Due to the difficulty in projecting future revenues and, hence, the allowable size of the LDRD program, some

LDRD Selection Process



funding is withheld from the normal project selection process. These corporate reserve funds are made available after the start of the fiscal year as Sandia's financial forecasts are better known. Projects that start after the fiscal new year are called "late-starts."

This year there was a special call for late-start proposals in S&T Strategic Objectives for "Engineering Excellence," ideas that support DOE's new nanoscience and technology initiative or that support Sandia's Science-Based Engineering for Trans-formation (SBET) initiative. One hundred seventy-four ideas for Engineering Excellence went directly to full proposals and 22 projects were selected.

There were 66 late-start projects that were reviewed and funded on a case-by-case basis. These were for opportunities that arose during the year and were considered to be too good to miss. For example, in "Large Atmospheric Explosions on Earth" (project 99868), the PI ran computer simulations of large atmospheric explosions and fireballs resulting from asteroid impacts on the Earth. Such an event may have been responsible for the mysterious origins of Libyan Desert Glass. The simulations were featured in summer 2006 BBC and National Geographic television documentaries and in Discover magazine. This provided an opportunity to publicly display Sandia's high performance computing capabilities, which are normally not visible due to the classification of the work.

In order to coordinate with the academic calendar, 51 University Collaborations projects are late-starts.

Sandia finished the fiscal year with 430 projects.

Note that there was a restructuring of Sandia's management during the summer of 2005. These changes will be reflected in the FY 2007 portfolio structure. The impact on the FY 2006 program was that the management of the IAs was assumed by a smaller group of managers called the Senior Steering Committee (SSC). The SSC was also instrumental in making program improvements this year.

FY 2006 Project Results

The NNSA requires Sandia to collect and report a number of measures of project productivity and quality, namely, cost, publications, intellectual property, and the number of post-doctoral appointees supported. Sandia collects these and additional metrics from the projects through the annual reporting process.

Cost:

The final cost of the FY 2006 program was $132M or 6.3% of laboratory operating costs. Historically, this cost will be viewed as an anomaly due to another change in the program, the financial loading of LDRD projects with the full overhead burdens of the Laboratories.

Selected Statistics on FY 2006 LDRD Projects

Total Funding ($M)	132
% of Total Laboratory Operating Costs	6.3
Total Projects	430
Average Project ($K)	305
Smallest Project ($K)	13
Largest Project ($M)	4.6
1st Year Projects	229
2nd Year Projects	131
3rd Year Projects	70
Defense Use (%)	18
Non-Defense Use (%)	16
Dual-Use (%)	66

According to Common Accounting Standards (CAS), LDRD projects are indirect (overhead) activities because they are conducted for the benefit of the company and their benefits are not attributable to particular customers. Consequently, LDRD projects have not borne the full overhead load structure prior to this year.

In the middle of FY 2006, Congress directed NNSA and the Labs to load LDRD projects with the full overhead burden structure as if they were direct projects. This resulted in, approximately, a one-third increase in project costs. Sandia implemented this change in the middle of the fiscal year and it was not made retroactive to the beginning of the fiscal year. Consequently, the reported cost of the FY 2006 program is “unloaded” for the first part of the year and “fully loaded” for the remainder of the year. The estimated FY 2006 cost using the “unloaded” FY 2005 structure would have been approximately $112M, 5.4% of laboratory operating costs, or using the “fully loaded” FY 2007 structure approximately $143M, 6.9% of operating costs. The FY 2007 cost will be “fully loaded.”

Publications:
In FY 2006, LDRD projects published 146 journal papers and made over 500 conference presentations. For the most recent five year period for which data is available, 2001-2005, Sandia LDRD projects produced 593 papers, approximately 20 percent of all of Sandia’s refereed papers, of which 72 percent were cited one or more times.

According to the Institute for Scientific Information (ISI), the LDRD papers with the greatest number of citations for each of the last five years are:

FY 2006 Project Productivity

Refereed Publications	389
Other Communications	360
Technical Advances	128
Patent Applications	37
Full Time Equivalent (FTE)	387
Average Project FTE	.9
Post-Doctoral Appointments	86
New Hires	6

2005: Fan, HY, et al., “Surfactant-assisted synthesis of water-soluble and biocompatible semiconductor quantum dot micelles,” in Nano Letters, 11 citations.

2004: Fan, HY, et al., “Self-assembly of ordered, robust, three-dimensional gold nanocrystal/silica arrays,” in Science, 42 citations.
2003: Tian, ZRR, et al., “Complex and oriented ZnO nanostructures,” in Nature Materials, 76 citations.
2002: Fleming, JG, et al., “All-metallic three-dimensional photonic crystals with a large infrared bandgap,” in Nature, 100 citations.

2001: Kim, SK, et al., “A gene expression map for Caenorhabditis elegans,” in Science, 206 citations.

In addition, the ISI ranks 325 institutions according to a metric that measures the impact of scientific papers on the scientific community by tallying the number of times published research is cited by other researchers. When Sandia’s LDRD papers are treated as a separate institution, ISI ranks Sandia’s LDRD related optics and acoustics publications third in the five years 2001-2005 (4.2 times the world average). LDRD papers were ranked among the top 20 in inorganic chemistry and nuclear engineering. Papers in applied physics/materials science were among the top 25.

Intellectual Property:
In FY 2006, LDRD projects filed 128 technical advances (TAs) and 37 patent applications. Over the last five years, LDRD projects have authored over 40% of all of Sandia’s TAs, almost 50% of the patent applications, and about 35% of the issued patents.

Two FY 2006 projects had exceptional intellectual property production, each producing 13 TAs and 2 patent applications: “Adaptable Software for Advanced Human/Computer Systems” (project 79738) and “Achieving a New Paradigm in Software Technology” (project 79883).

Post-docs and Other People Metrics:
Over 2000 laboratory staff provided support for LDRD projects in FY 2006, the equivalent of about 440 FTEs. Eighty-six of those were post-doctoral researchers. LDRD typically supports about half of Sandia’s post-doc population. Historically, more than half of the post-docs and technical limited term employees (LTEs) that have converted to full-time staff were supported by LDRD.

More than 200 hundred students were supported, most attending universities that are strategically important to Sandia.

LDRD projects were responsible for hiring new employees, students, and contractors to bring critical new skills to Sandia. For example, “Microscale Immune Study Laboratory (MISL)” (project 95215) hired five researchers trained in biological sciences.

Awards and Leadership:
Awards and other prominent recognition help to establish Sandia as an apparent leader in science and technology.

One of the two R&D 100 Awards won by Sandia in FY 2006 was related to an LDRD project (“Algorithmic Support for Commodity-Based Parallel Computing Systems,” project 26516, FY 2001-2003). The Compute Process Allocator is a computer algorithm technology that increases processing efficiency on massively parallel supercomputers. Developed in conjunction with colleagues at the State University of New York and the University of Illinois, the CPA’s principal developer is Vitus Leung, along with Kevin Pedretti and Cynthia Phillips. The CPA was licensed to Cray Inc. in 2005.
LDRD made the covers of three journals in FY 2006.

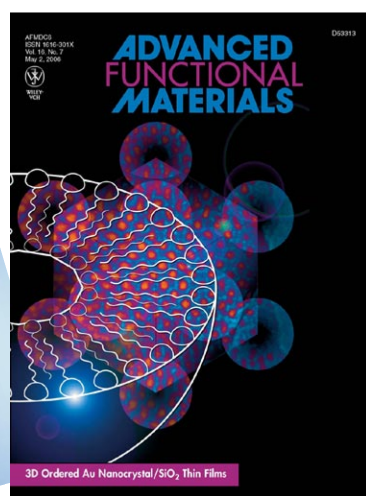
Advanced Functional Materials, Vol. 16 No. 3, 2006, features art from the paper “Sequential Nucleation and Growth of Complex Nanostructured Films,” related to “Development of Nanostructured and Surface Modified Semiconductors for Hybrid Organic-Inorganic Solar Cells” (project 93564).

Advanced Functional Materials, Vol. 16, No. 7, 2006, features art from the paper “Three-Dimensionally Ordered Gold Nanocrystal/Silica Superlattice Thin Films Synthesized via Sol-Gel Self-Assembly”, related to “Engineered Conjugated Molecule-Linked Metal Nanocrystal/Silica Arrays for Integrated Chemical Sensor Platforms” (project 104953).

JOM, September 2005, features art from the paper “Nanotribology of Anti-Friction Coatings in MEMS,” related to “Nanoparticle Flow, Ordering and Self-Assembly” (project 93529).

Collaborations:
External collaborations are important to Sandia in various ways. University collaborations, for example, help to attract students to Sandia and industry collaborations help with technology transfer. In FY 2006, 189 projects collaborated with universities, industry,

93564 – Julia Hsu, Development of Nanostructured and Surface Modified Semiconductors for Hybrid Organic-Inorganic Solar Cells



104953 - Hongyou Fan, Engineered Conjugated Molecule-Linked Metal Nanocrystal/Silica Arrays for Integrated Chemical Sensor Platforms

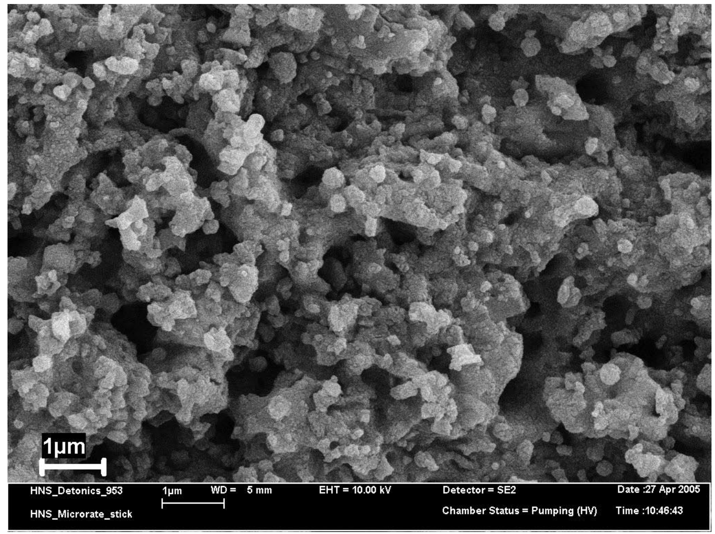
93529 – Gary Grest, Nanoparticle Flow, Ordering, and Self-Assembly or other government laboratories. There were 285 separate collaborations with universities, 41 collaborations with industry, and 47 collaborations with other government labs.

Further Work:
Successful R&D leads somewhere, perhaps to sponsored R&D or perhaps to more LDRD. In FY 2006, the PIs reported that 107 projects led either to a new sponsored project, an increase in funding for an existing sponsored project, or a lead for a prospective sponsored project. Eight projects led to cooperative research and development agreements (CRADAs). Fifty-six projects led to another LDRD project.

Impact of Technical Risk:
We collected a new metric in FY 2006, a description of the impact of technical risk on the project during the year. While risk is difficult to measure in advance, we are able to assess it retrospectively and we now ask the PIs what impact technical risk had on their projects in the current fiscal year.

Only 39 percent of the projects reported “No Impact” this fiscal year, while 42 percent reported one of three kinds of adverse impact: Twenty-six percent reported changes to their R&D approach, e.g., changes in project tasks, milestones, cost, or schedule. Twelve percent reported needing to scale back their R&D goals or their problem to be solved. Four percent reported their originally proposed R&D goals/ objectives could not be met or the originally proposed problems could not be solved and it was decided to terminate the project or to redefine the purpose of the project and go in a new direction.

Adverse impacts are typically undesirable in the R&D directed by DOE, NNSA, and other sponsors. But where an external sponsor might terminate a project for negative results, Sandia may instead redirect and rejuvenate an LDRD project by changing its plan, scope, or goals. This allows researchers to continue to pursue R&D paths with potential value and protects the already committed investment in R&D. This approach is a hallmark of the LDRD program and encourages researchers to stretch beyond their current



Femtosecond laser micromachining of explosives generates submicron particles

capabilities.

The impact of technical risk is not always negative. Twenty-five percent of the projects reported some sort of positive impact of the technical risk. (Some projects reported both positive and negative impact.) For example, “Micro- and Mesoscale Detonics of Explosives” (project 79871) discovered that femtosecond laser micromachining of explosive material generates submicron particles. This led to additional characterization of the nanoparticles in collaboration with Los Alamos National Laboratory and New Mexico Institute of Mining and Technology and the filing of several technical advances.

We are ceasing to collect two metrics that had the unintended consequences of discouraging risk taking. Some projects interpreted the tracking of milestones as implying that the project should not deviate from the R&D plan. Likewise, for some projects, tracking whether a research hypothesis is proved implied that only positive results are desired.

Future Program Directions
At the time of this writing, the FY 2007 program is under way and we are planning for FY 2008. In the immediate future, we have three areas of concentration for program improvements: investment strategy, program efficiency, and outcome management. Investments in LDRD projects must be made

strategically. We are making efforts to better align the Investment Areas with the Laboratories' strategy. This is reflected in the FY 2007 program structure. We are also improving the IA Calls to better express their strategies.

The program must be efficient. We continue to make internal changes for efficiency, for example, improving the idea descriptions so that better decisions can be made at the idea phase resulting in less time spent on unsuccessful full proposals.

The outcomes of R&D must be managed. Outcomes contrast with the ordinary outputs of LDRD in the following way: Outputs are the immediate, tangible products that are easily attributed to a particular project, for example, research results, publications, patents, and so on. Outcomes, on the other hand, are less immediate, less tangible, and often difficult to attribute to a particular project.
Examples of LDRD outcomes include leadership in science and technology, Sandia's reputation, program growth, technology transfer, and other strategic objectives. An example of outcome management is the creation LDRD Award for Excellence and LDRD Day: the nomination process reinforces management review of completed projects and increases the chances for impact. We are investigating tools to improve outcome management, including structure, process, and assessment.

FY 2006 has been a significant year for Sandia's LDRD program. This Annual Report provides a mere snapshot of a dynamic program. The reader is encouraged to dig into this report to learn more about the projects that have been mentioned and others. More information can be obtained by contacting the LDRD Program Management Office.

Science, Technology, and Engineering

The LDRD Program helps Sandia maintain its position as a world leader in science, technology, and engineering (ST&E). The objective of ST&E investments is to create the new science, technology, and engineering essential to providing innovative solutions to national security problems.

ST&E projects seek new understanding across multiple disciplines through the mission-focused pursuit of advancing science and technology. They support science-based engineering and product realization related to weapons, nonproliferation, energy, critical infrastructures, and environmental missions of the Department of Energy and other federal agencies. Areas of ST&E investments include:

- Materials Science and Technology
- Advanced Manufacturing
- Advanced Components and Certification Engineering
- Pulsed Power Sciences and High Energy Density Sciences
- Engineering Sciences
- Electronics and Photonics
- Computational and Informational Sciences
- ST&E Strategic Objectives
- Biotechnology

Projects under the Materials Science and Technology umbrella focus on developing innovative materials and materials processing concepts that enhance Sandia's ability to steward the nuclear weapons stockpile and minimize or mitigate possible national security threats. This focus includes understanding predictive performance, aging, and reliability of new and existing products and components.

Pursuits in Advanced Manufacturing support the advancement of science and technology to enhance Sandia's capabilities to fully understand and model manufacturing at the system, operation, and process levels. Projects aim to develop innovative new process technologies and techniques for product realization.

Advanced Components and Certification Engineering investments focus on advanced development and technology integration, and enable exploratory efforts centered on weapons systems, subsystems, and component capabilities. Projects also work toward lighter, less expensive packaging that quickly and efficiently supports greater functionality within a constrained testing environment.

The ST&E investment category also supports the study of innovative ideas within the Pulsed Power and High Energy Density Science fields that could lead to the development of experimental, test, computational, and analytical capabilities for application to stockpile stewardship, basic science, industrial competitiveness, military technologies, and future energy production.

Project teams are encouraged to perform multidisciplinary work that builds on our differentiating strengths to achieve integrated processes and technologies. Specific applications and missions include inertial confinement fusion, hostile nuclear environment survivability, electromagnetic propulsion and launching, defeat of biological agents, and inertial fusion energy.

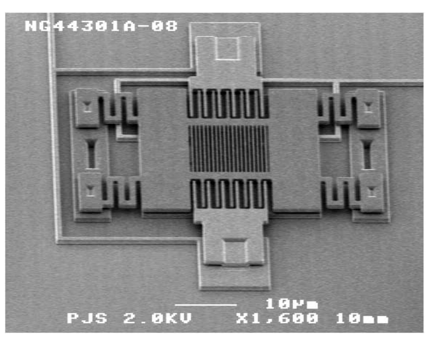


Figure A

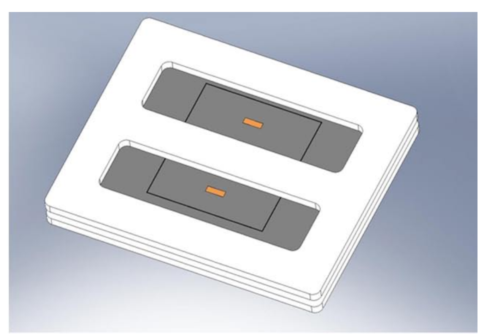


Figure B

Project 67023 Nano-g Accelerometers Using Nanophotonic Motion Detection System

Optical nanophotonic sensor (Figure A) used to build a new class of in-plane, highly-sensitive (10 ng/√Hz), ultraminiature (< 1 cm³) accelerometers in silicon (Figure B).

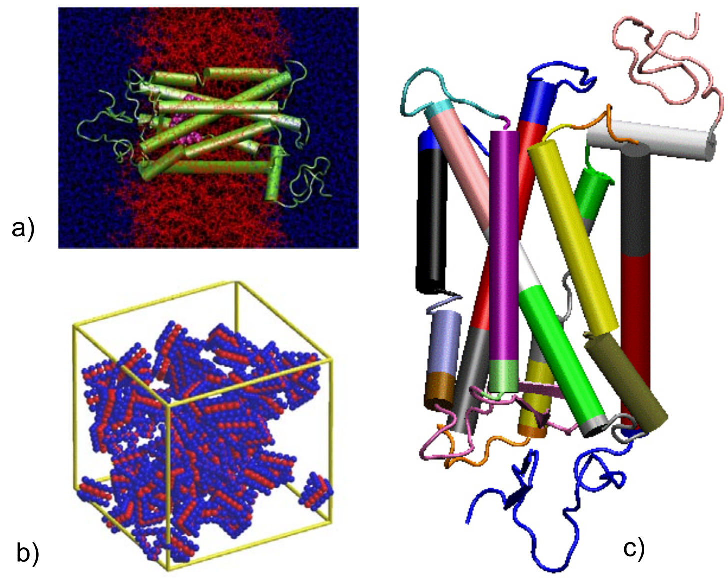
Investments in Electronics and Photonics include sensor concepts that advance semiconductor science and technology and seek to provide the capability to realize small, low-power, highly integrated microsystems for national security applications.

Project 67023, Nano-g Accelerometers Using Nanophotonic Motion Detection System, for example, fabricated and characterized surface micromachined diffractive optical grating structures that modulate light when moved.

The team developed subwavelength grating-based accelerometers that can sense accelerations as small as 10 ng/Hz1/2. No other devices or products come close to this level of sensitivity. The high sensitivity and small size of these devices enable a variety of new applications in the areas of seismology and navigation.

Engineering Sciences research advances the understanding of engineering phenomena through the development and use of computational, experimental, and theoretical capabilities. Fundamental engineering sciences research focuses on innovative technologies and multidisciplinary capabilities that provide unique and discriminating strengths.

Research in Computational and Information Sciences supports national security missions by developing capabilities for applications that include nuclear weapons safety and reliability, design, manufacturing, electrical systems, microelectromechanical sytems (MEMS), biotechnology, and homeland security.



Project 67017, Substructured Multibody Molecular Dynamics

a) 150-ns biomolecular simulation of rhodopsin photoisomerization shows early steps in visual sensing. b) Simulation of functionalized nanoparticle self-assembly. c) Seamless multigranular substructured molecular dynamics now possible using Sandia's LAMMPS code.

One effort in this area was Project 67017, Substructured Multibody Molecular Dynamics, where the team developed advanced molecular simulation capabilities, especially the ability to perform substructured multibody molecular dynamics simulations.

The team demonstrated that the enhanced software produced correct thermodynamic, kinetic, and structural characteristics for a simple alanine dipeptide test problem, using both an atomistic and rigid body model, and for other test problems, including alkane systems, a RuBisCO protein system, a rhodopsin protein system, and a nanoparticle self-assembly system.

The software has been released and the tools are in place to perform coupled rigid body simulation within the molecular dynamics framework.

Another key strategic goal of ST&E investments is to prepare for the emerging crises in global energy supply, terrorism, and proliferation of weapons of mass destruction. Small levels of highly targeted funds are invested in Science and Technology Strategic Objectives to promote advanced and innovative research and development in support of long-term ST&E needs.

The ST&E investment area continues to support research that pushes beyond Sandia's traditional core nuclear weapons mission, specifically in biotechnology and chemical and earth sciences.

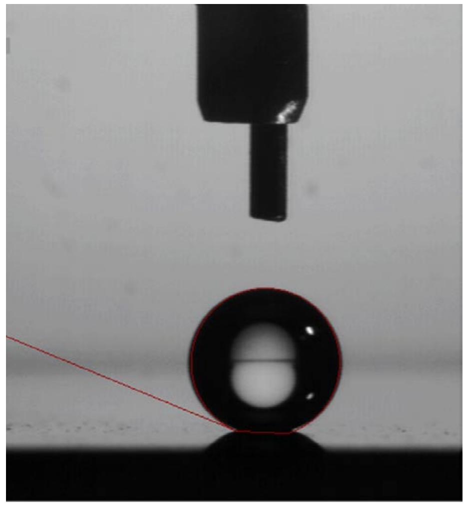
Investments in Biotechnology seek to understand biological processes and their products to harness and leverage these processes for national security applications such as bioterrorism, nonproliferation of biowarfare technologies, and energy security.

Endeavors in Chemical and Earth Sciences explore research in chemical signature characterization for stand-off detection, remote sensing of geologic and chemical properties, fate and transport in natural systems, carbon cycle science, geophysical imaging of the subsurface, simulation of earth materials, combustion science, and laser detection of reactive species.

Experiments completed as part of Project 73185, Superhydrophobic Surface Coatings for Microfluidics and MEMS, are adding to the body of knowledge regarding the conditions that cause liquid droplets to roll rather than flow/slide on a surface and how this "rolling transition" influences the boundary condition describing fluid flow in a pipe or a microchannel.

Rolling of droplets is important, for example, for aerosol collection strategies, because it allows trapped particles to be concentrated and transported in liquid droplets with no need for a predefined/micromachined fluidic architecture.

The team discovered that cavitation is a source of long-range hydrophobic interactions; is a consequence of, and thermodynamically consistent with, the properties of confined water; and might play a role in protein folding phenomena and other diverse phenomena that occur under water.



Project 73185, Superhydrophobic Surface Coatings for Microfluidics and MEMS

Water droplet on superhydrophobic silica surface

Advanced Components and Certification Engineering

Next-Generation High-Voltage Switches for Capacitive Discharge Firing Systems

79868

F. R. Trowbridge, K. A. Peterson, C. A. Walker, B. Wroblewski, T. A. Friedmann

Project Purpose

High-voltage switches with high peak current capability and precise, repeatable performance are essential components in capacitive discharge systems. Current firing sets rely on custom-triggered vacuum systems as switches.

This project is focused on generating, developing, and evaluating switch designs that provide comparable performance to existing systems while exploiting advances in microfabrication and batch manufacturing. Using such technologies allows us to develop switches with characteristics that can be tightly controlled and made consistent within batches. Cost savings, reproducibility, and reduction in hand manufacturing are intended benefits of this research.

This year, we demonstrated the feasibility of using microfabrication techniques combined with conventional vacuum processing technology. We successfully built and tested high-voltage switches based on conventional ceramic substrates with through-hole connections. Thin film processing techniques created triggering layers on top of the ceramic, using masking and pulsed-vapor deposition. These switches showed good voltage standoff and current conduction capability. They also conditioned much like a conventional Sprytron; shot lifetimes ranged to 100 shots.

Next year, we will focus on exploring several advanced concepts for switch development. Using plasma etching, trenches within the alumina substrate will allow for a thicker carbon film reservoir to be created. This reservoir will lengthen shot lifetime by serving as a source for the redeposition of carbon onto the trigger film. We will explore other designs that do not require a metal via through the ceramic.

Leakage through the via frequently degraded the vacuum within the switches, rendering the part unacceptable. Other advanced development will involve using material other than carbon for the trigger film. Ferroelectric materials show strong electron emission when pulsed.

FY 2006 Accomplishments

Demonstrated and characterized switch operation at voltage and current levels compatible with existing nuclear weapon firing systems

We successfully demonstrated the operation of a ceramic-based microfabricated switch using Sprytron firing sets. The parts tested at voltage and current levels within the range needed for existing firing systems.

Continued process refinement for graphite-based trigger solutions

We continued to refine our process by using different deposition and etching methods, and successfully created functional carbon trigger films using a pulsed laser deposition method. We are pursuing reactive ion etches to etch alumina and improve our trigger design.

Continued process refinement for the braze closure and packaging assembly

We overcame difficulties presented using conventional brazing, and refined the braze mixtures, volumes, and pressures to achieve a functional switch. Many parts leaked through the via interconnect to the carbon trigger film; we are evaluating configurations that will improve part hermeticity.

Developed and evaluated switches that use boron-doped diamond for the trigger element
We delayed this approach in order to explore the use of ferroelectric emission materials, which promise to create vacuum switches with extremely high shot lifetimes. Processing ferroelectrics is much simpler and less expensive than processing diamond.

Modeled and simulated device operation and correlation with observed switching performance
We used static electromagnetic models of the vacuum switch to design working switches. We presented pre-breakdown models at a conference in April and will continue to develop them.

Significance

This project is a multidisciplinary effort involving several Sandia centers and a collaboration with Auburn University. The effort will benefit the DOE/National Nuclear Security Administration/ DP Advanced Initiation Program by enhancing firing system pulse discharge technologies through collaborative research in the areas of integrated product and process design and advanced manufacturing, and advanced modeling and analysis applied to high-voltage switch technology.

The low cost and small size of the switch will enable its use in non-DOE platforms as well, in particular, high-voltage Department of Defense fuzing systems.

Intelligent Fuzing for Hard Target Defeat

79869
R. McEntire, D. W. Kelton, T. G. Carne, M. S. Allen

Project Purpose

The purpose of this project is to develop algorithms for use in advanced fuzing applications for the real-time detection of complex hardened targets. This project also addresses a need to characterize real targets for penetration events. More precise target characterization enables a better assessment of the probability of success of attacking a target.

It has long been known that acceleration records of penetration events can be used to characterize targets. However, the challenge for target characterization in impact applications is separation of the desired rigid body deceleration from the confusing oscillatory modes of the weapon system itself.

The approach taken in this method is to remove the modal "noise" on the acceleration record by the sum of weighted accelerations technique (SWAT). SWAT uses a small number of sensors along weapon axes to determine (in the laboratory) the system's modes. Once the modes are known, a weighting function is developed through laboratory testing and is designed to cancel the vibratory content produced by modal vibration of the system. The resulting algorithm will provide real-time acceleration without the filtering time delay created by traditional filtering techniques.

Fuzing systems employ an array of sensors and locking mechanisms to maximize safety and prevent inadvertent firing. However, the more sensors that are included, the more costly the device becomes to build and maintain. Therefore, a secondary purpose of this project was to try to determine the minimum number of sensors needed for the SWAT technique to reliably sense and detect impact in a penetration environment.

FY 2006 Accomplishments

The second year objective was to improve upon the previous year's SWAT system accomplishments by recommending sensor placement strategies for a penetrator system that will be evaluated in actual penetration environments (this project had neither the funding nor objective of actual flight hardware).

In order to best simulate the type of sensors that could be included in a minimalistic fuze system, we selected five sensors – one axial accelerometer and four strain gauges – and mounted them circumferentially about the forward diameter of the penetrator.

We instrumented a scaled penetrator, similar to those in use for high-velocity penetration experiments, with one triaxial accelerometer (note that this is more than the single axial accelerometer that might be used in a flight fuze) and four strain gauges equally spaced about the penetrator diameter. We applied a short impulsive force to the penetrator nose with a force-gauge instrumented hammer.

We now know the input signal and can use it to compare the output signal received from both the accelerometers and the strain gauges. We created a frequency response function (FRF) and a Fourier transform of both the acceleration and the strain responses. These plots showed the first and second bending modes of the penetrator as well as the first axial mode.It is the axial mode that we wish to eliminate from the deceleration-time signal. However, noise created by effects picked up from the first and second bending modes can also be detrimental.

Since the four strain gauges are installed on opposite sides of the penetrator body, if a bending mode is induced, one strain gauge will be in tension, while the one on the opposite side will be in compression. Thus, summing them together should net an effective bending strain of zero.

We obtained the FRF sum of the four strain gauges and compared it to that of a single strain gauge. There was marked improvement in the bending mode response.

Significance

A reliable impact detection technique is the desire of any hard target fuze. The SWAT technique may eliminate penetrator modal motion from the deceleration time history. This would allow for more accurate representation of the target characteristics by avoiding the weapon internal dynamics. However, SWAT works best when the sensors have a maximum distribution about the center of mass.

A fuze typically must house and contain the sensors it uses within its own body, which may minimize the separation distance that can be achieved. Still, the technique has shown promise as a way to remove internal modal motion from an acceleration signal and improve detection reliability.

However, there may be a need to include either lateral accelerometers (such as a triaxial accelerometer) or an additional set of four strain gauges to effectively limit the bending modes seen on the sensors. This may be an area of continued future research.

A secondary result of the experiments was the comparison of the measured strain-gauge “force” against that recorded by the calibrated hammer. When compared, the force extracted by the strain gauges laid nearly on top of the force recorded by the impulse hammer.

This may imply that a set of strain gauges can be used in penetration experiments to extract the force applied to the nose during impact – especially for lower velocity impacts (<1000 ft/sec) where the frequency content of the impact force rolls off at higher frequencies.

Refereed Communications

M.S. Allen, T.G. Carne, “Comparison of Inverse Structural Filter (ISF) and Sum of Weighted Accelerations Technique (SWAT) Time Domain Force Identification Methods,” in *Proceedings of the 47th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics, and Materials Conference*, pp. 1-24, May 2006.

A New, Cost-Effective Solution to Provide Radiation-Hardened Materials for Nuclear Weapons

79870

P. J. Cole, V. K. De Marquis, L. F. Bieg, J. L. Lenhart

Project Purpose

Polymeric materials are used throughout the nuclear weapons complex for a variety of applications. In typical applications, the polymer is a critical material for electronic devices. Many of these devices have a radiation tolerance requirement; unfortunately, common polymeric materials are typically not radiation tolerant.

Radiation exposure generates electron-hole pairs in the polymer. The mobile electrons generate radiation-induced conductivity (RIC), which can degrade device performance. This project will establish the scientific foundation required to understand RIC in common polymeric materials and how to reduce RIC.

Since electron-hole pairs are generated during radiation exposure, incorporation of small molecule electron traps or electron donors offers a mechanism to improve radiation tolerance and reduce RIC. In order to incorporate these additives in polymeric materials, post-processing strategies must be developed. A common mechanism for incorporating small molecules in polymeric films is to immerse the film in a solvent bath containing the dopant and rely on dopant diffusion into the polymeric materials.

To establish the scientific foundation for producing radiation-tolerant polymeric materials, the specific objectives of this project are to:

1) identify classes of small molecule dopants that are effective at reducing RIC in polymers
2) understand the factors that control the doping process and the resulting RIC in polymer films
3) provide a fundamental framework for understanding the thermodynamic and kinetic limitations of small molecule incorporation in organic films that can be extracted to meet broader material needs in the nuclear weapons complex.

To demonstrate the practical implications of this scientific foundation, an additional focus will be to develop radiation-tolerant polymer dielectrics for high-voltage capacitors used extensively in the weapons complex.

FY 2006 Accomplishments

We had four major accomplishments this year.

Identified several small molecule dopants that can effectively improve radiation performance in polymeric films

Electron trapping molecules were the most effective at reducing RIC in polymeric materials. The typical structure of the electron traps we tested contained a central aromatic structure with pendant electron withdrawing groups. Molecules that contained nitro functional aromatics were most effective at reducing the RIC in the polymer. Ongoing research is aimed at investigating the links between the electron trapping capability in the small molecule and the concentration of that molecule in the polymer to the resulting RIC.

Developed a solvent-assisted process for incorporating the dopant into the polymer

In order to incorporate the small molecule dopant into the polymer, we developed a solvent-assisted process in which the dopant is dissolved into a solvent at elevated temperatures. The polymer is immersed in the solvent solution, and the dopant diffuses into the polymer film. Initial research determined that to effectively dope the polymer film, the solution temperature must be higher than the polymer glass transition temperature.

Investigated critical factors that control the doping process

The polymer-solvent interfacial region was determined to be a critical factor in the doping process. In particular, the dopant concentration in the polymer was typically substantially higher than in the bulk solution concentration, indicating a segregation of dopant to the polymer interface. In addition, when the polymer surface chemistry was modified by a ultraviolet-ozone exposure, the quantity of dopant in the polymer film changed, further indicating that the polymer-solvent interface is a controlling factor.

The most effective type of solvent for doping the polymer films can be different for each dopant-polymer combination. In addition, two solvents with similar solubility parameters might have different effective doping abilities, indicating that the solvent solubility parameter is not a controlling factor.

Investigated fundamental links between the molecular structure of the small molecule dopant, the incorporation of this dopant into a polymer, the dopant concentration, and the resulting RIC
More work is required to develop the fundamental understanding associated with this process. In particular, we noticed that polar functional groups off the ring were important for enhancing the doping in Mylar films. This is likely due to enhanced surface adsorption of the dopant on the polar polymer. In addition, nitro functional groups off the ring were extremely effective at reducing the RIC.

Significance

The project is extremely significant to Sandia's weapons mission, since many devices have a radiation tolerance requirement. By understanding the factors that control RIC in polymeric materials, as well as strategies to reduce that conductivity, improved materials and devices can be designed for future weapons and life extension programs.

The project also has significance for nonweapons programs, such as satellite and space applications, which also can have RIC issues.

One major driver for this project is that expensive and heavy metallic-based radiation shielding and processing strategies can be potentially eliminated if the intrinsic device materials are radiation tolerant. Therefore, this project will likely help to reduce materials and manufacturing cost for future components both internal and external to Sandia.

In addition, the understanding of kinetic and thermodynamic factors that control small molecule incorporation in polymeric films will have a profound impact on the commercial polymers sector, since dopants are used extensively in polymers to control processability, color, and optical properties.

Conductivity in organic materials is poorly understood but has a profound impact on many emerging technologies such as sensors, fuel cells, and biomedical applications, which exploit electronic or ionic transport in polymers. Understanding the chemical and physical factors that control RIC in polymers will enable materials advances in these emerging fields.

Other Communications

P.J. Cole and J.L. Lenhart, "Radiation-Hardened Materials for Use as Dielectrics," presented at the 36th Annual Polymeric Materials Aging and Compatibility Conference (PolyMAC), Lawrence Livermore National Laboratories, June 2006.

Micro- and Mesoscale Detonics of Explosives

79871
A. S. Tappan, E. J. Welle, A. L. Brundage, J. A. Palmer, S. K. Marley

Project Purpose

The primary goals of this project are to generate critical performance data that can be used by conventional and microexplosive train designers to better understand margin of designs, as well as to provide data that are useful to the modeling community. These data can be dramatically different in nature as modelers typically need performance data for the materials in question, but they also need well-specified boundary conditions to generate appropriate models.

Microscale effects typically manifest as failure diameters, detonation wave spreading, shock-to-detonation transition (SDT), and deflagration-to-detonation transition (DDT) distances, where failure diameters, SDT, and DDT characteristics affect initiation performance of conventional and microexplosive trains. Characteristics of the explosive that affects those traits include the chemical nature of the explosive as well as its crystallographic and particle nature. Consequently, we are studying primary and secondary explosives with differing particle sizes to more precisely quantify the effects of those variables.

The data we are collecting include failure diameters, shock-to-detonation lengths and deflagration-to-detonation lengths for primary and secondary explosives. To generate these data, we employed creative and unique methods to produce explosive charges of appropriate size scales.

For primary explosives, we developed a fluid-based preparation technique in which a dispersion of explosive particles is drawn into a small-diameter capillary (as small as 50 microns). Through careful control of the dispersion concentration, settling conditions, and filling methodology, we are able to produce homogeneous, high-quality samples.

For secondary explosives, we used a novel method to produce explosive fixtures by using femtosecond laser machining (FLM) of explosive charges.

As with many new techniques, unexpected results and benefits were generated. Preliminary analysis of FLM by-products showed that nanoexplosive materials were generated during FLM. This revelation has led to technical advances as well as collaborations with New Mexico Institute of Mining and Technology and Los Alamos National Laboratory (LANL) to characterize the material. These two production methods provide samples for high-fidelity explosive characterization experiments to provide useful data for both designers and modelers.

This work is truly appropriate for LDRD, as it seeks to identify critical characteristics of energetic materials that impact performance of such devices. We seek to identify mesoscale parameters that have high consequence on initiation performance and failure characteristics with the goal of defining critical design information for micro- and macroexplosive trains, as well as generate data that is of great value to the modeling community. In essence, it represents a science-based approach to enable understanding-based engineering designs.

FY 2006 Accomplishments

Many of this year's accomplishments have come in the form of data generated that better define explosive properties at submillimeter length scales. The explosive materials we investigated are HNS, CL-20, lead azide, and silver azide. HNS and CL-20 are secondary explosives, while the two azides listed are primary explosives. All materials under evaluation are expected to exhibit favorable characteristics for integration into miniaturized explosive trains, but are also of great interest for conventional systems.

A large portion of our efforts were centered around proving a new diagnostic technique for determining critical or failure diameter for explosives using streak camera photography and micromachined rate sticks. These rate sticks are generated by using the femtosecond laser to machine-press pellets much like a traditional lathe would shape a cylinder of metal. The method used to test these explosive fixtures is to initiate the charge at the base using a chip-slapper to ensure precise experiment timing and then track the detonation wave velocity up the length of the rate stick.

We conducted a number of experiments on submillimeter rate sticks of HNS and CL-20.Through a combination of simulations and experiments, we designed an experimental configuration that allows precise measurement of the rate stick reaction velocity but is free of any obscuring effects from the necessarily larger base explosive pellet. This configuration involves machining a taper in the base pellet up to the point of the rate stick and potting this taper with silicone rubber, thus providing sufficient input and mechanical strength without allowing base pellet detonation products to obscure the rate stick. We conducted experiments on rate sticks with diameters as small as 187 microns.

An unexpected result of the FLM of explosives was finding that the machined by-products were still explosive yet had a different morphology. Scanning electron microscopy of the machined powder revealed that nanometer scale particles were generated. This finding sparked a collaborative study with New Mexico Tech and LANL, with independent verification of the chemical nature of the products using infrared spectroscopy and electron beam diffraction.

In addition, we developed a method for reliably producing lead azide and silver azide charges in sub-millimeter geometries using capillary action to draw an energetic material dispersion into a microcapillary. Streak-camera experiments determined deflagration-to-detonation properties of both lead azide and silver azide in charge diameters of 200, 100, and 50 microns. Initiation was accomplished by direct irradiation with a Nd:YAG laser. This fundamental work is being performed by our graduate student for his master's thesis at Texas Tech University.

Significance

This work will enable the design of next-generation microscale energetic weapon components using MEMS (microelectromechanical systems) technologies. Such components will be smaller, safer, and find greater use in new weapons systems. In particular, such low-mass components may be key to successful penetrator designs or gun-launched munitions.

The technologies we developed for the creation of submillimeter explosive charges and diagnosis of submillimeter explosive phenomenology are unique and applicable to future research. Our discovery of a new technique for the production of nanometric explosive particles is significant and may enable further miniaturization. In addition, critical insight into transient phenomena that control conventional and micro-ordnance device functionality is of fundamental importance for miniaturization needs.

Additional measures of quality of work come in the form of publication of this work in The 13th International Detonation Symposium, which is the premier venue for fundamental explosives research.

Refereed Communications

E.J. Welle, A.S. Tappan, R.J. Pahl, J.A. Palmer, and R.E. Muenchausen, "Diameter Effects on Detonation Performance of HNS and CL-20," in *Proceedings of the 13th International Detonation Symposium*, July 2006.

S.P. Madden, A.S. Tappan, P.C. Jung, S.K. Marley, E.J. Welle, and R.J. Pahl, "Rapid Data Analysis Methodologies for Streak Camera Images: Measurement of Detonation Velocity and DDT Distance of Lead Azide at Submillimeter Diameters," in *Proceedings of the 13th International Detonation Symposium*, July 2006.

Other Communications

S.M. Harris, E. Welle, J.A. Sanchez, R.E. Muenchausen, and C. Hockensmith, "Analysis of Femtosecond Laser Generated Nanoenergetic Particles," presented at Defense Manufacturing Conference, Orlando, FL, November 2005.

J.Palmer, E.J. Welle, and A.S. Tappan, "Advances in Ultrashort Pulse Laser Micromachining of Explosives: New Products and Processes," presented at DEPS Ultrashort Pulse Laser Materials Interaction Workshop, Boulder, CO, September 2006.

Ion Neutron SIMulation - INSIM

79874

G. Vizkelethy, H. P. Hjalmarson

Project Purpose

Exposure to radiation damages electronic components such as bipolar junction transistors (BJTs) and, as a result, significantly degrages their performance. In certain environments, the radiation consists of short, high-flux pulses of neutrons. These neutrons cause displacement damage in the base region of the BJTs leading to the degradation of their amplification gain.

To further complicate matters, the defects anneal with a complex temporal behavior that depends on the atomic nature of the defect and the local electron density. This "transient annealing" leads to a time-dependant behavior of the amplifier gain, which also must be considered when analyzing and certifying device performance. Devices and circuits are usually tested in pulsed nuclear reactors; however, the availability of these facilities has decreased dramatically in the past few years, and the future of the remaining few is uncertain.

The purpose of this project is to simulate the effects of neutrons by directly implanting high-energy Si (or other) atoms into the active regions of devices to simulate displacement damage effect.

Neutrons affect semiconductors such as Si by causing atomic displacements of Si atoms. The first displacement is caused by the nuclear scattering of the neutron itself, producing a recoil Si atom, which continues through the lattice, causing many more displacements via a collision cascade. This is plausible because it is the collision cascade, caused by neutron-recoiled energetic Si atoms, that causes most of the damage to the Si semiconductor crystal.

Our intent was to develop a new technique using easily available particle accelerators to provide an alternative to pulsed nuclear reactors to study the transient annealing in various devices and qualify them against radiation effects.

FY 2006 Accomplishments

Measured the fluence of the ions incident on the BJTs

Fluence is one of the most important parameters of these experiments. In the case of neutrons, it is easy to measure the fluence since the pulses are highly reproducible, and it is enough to characterize the environment a few times. Also, the neutron beam is large, and it is possible to place real-time monitors into the same neutron environment where the test devices are.

On the other hand, in the case of the ions, the beam is focused into a small spot to achieve the required high fluences. In most cases this spot is hardly larger than the device and does not allow us to monitor the radiation environment in real time.

We developed a method to characterize the ion beam irradiation environment just before and after the ion beam pulse actually hits the devices, giving us a way to estimate the ion beam fluence and quantify its uncertainty.

Increased particle flux

We installed a new ion source, which resulted in a tenfold increase in the particle flux for certain ions. This increase was necessary to reach fluence levels comparable to those achieved in pulsed nuclear reactor in equally short time.

Calculated the displacement damage

We performed Monte Carlo calculations to calculate the displacement damage in the base region of the BJTs in order to compare the effect of different ions on the gain of the BJTs. The results of these calculations, compared to the neutron damage kerma, gave a starting point for the damage equivalency studies.

Irradiated BJTs to establish damage equivalency

We performed irradiation on 2n2222 and 2907 BJTs with various ion beams (different energies and different ion species) at different fluence levels to establish damage equivalency between neutrons and ions. The calculated damage ratios ($k = (1/G-1/G_0)/$ fluence, where G and G_0 are the final and initial gains of the transistor) agreed with the displacement damage ratios calculated using the Monte Carlo codes for heavy ions and neutrons.

Discovered nonlinearity

We discovered a nonlinearity in the inverse gain versus fluence for light ions (hydrogen and helium) at low fluences. This nonlinearity is a result of the ionization effect in the field oxide of the BJT and it is being investigated further. We also found that the inverse gain degradation becomes nonlinear for extremely high fluences when the low-doped collector of the BJTs is being compensated because of the large displacement damage.

Compared to pulsed nuclear reactor-irradiated BJTs

We performed DLTS (deep level transient spectroscopy) measurements on BJTs irradiated at different fluence levels and compared them to the ones irradiated in pulsed nuclear reactors. The DLTS measurement gives information about the defect levels and number of defects in the collector-base junction. The DLTS spectra were quite similar for the neutrons and heavy ions at lower fluences while there were differences for lighter ions.

Significance

As of September 30, 2006, the Sandia Pulsed Reactor (SPR-III) was shut down permanently. SPR-III was the main test site for the study of transient annealing in BJTs caused by displacement damage from short, high-fluence neutron pulses.

This project demonstrated that high-energy heavy ions can be used to simulate MeV neutron damage in BJTs. The results of this project are being integrated into the QASPR (Qualification Alternatives to SPR-III) effort. QASPR, a many-year multimillion-dollar program, is considered a vital project for Sandia and one of its largest. These results also raised the basic understanding of the damage equivalence between different radiation environments and provided data to modelers in the QASPR program.

Refereed Communications

E.S. Bielejec, G. Vizkelethy, D.B. King, and N.R. Kolb, "Damage Equivalence of Heavy Ions in Silicon Bipolar Junction Transistors," to be published in *IEEE Transactions on Nuclear Science*.

Other Communications

E.S. Bielejec, G. Vizkelethy, R. Fleming, and B.L. Doyle, "Radiation Effects in Silicon BJTs Using Both Active Gain and DLTS," presented at 19th International Conference on the Application of Accelerators in Research and Industry, Fort Worth, TX, August 2006.

A Miniaturized mW Thermoelectric Generator for Nuclear Weapons Objectives: Continuous, Autonomous, Reliable Power for Decades

79875

C. A. Apblett, T. L. Aselage, M. W. Moorman, R. P. Manginell, D. Ingersoll

Project Purpose

The intent of this project was to discover the critical requirements for the miniaturization of a thermoelectric power device. Based upon thermal modeling and microfabrication techniques, our goal was to demonstrate a prototype system generating 1mW in as small a volume as possible. Another goal was to use a nuclear power source and minimize both the size of the material used as well as the amount of shielding needed for the source to be considered reasonably safe. We simulated the source with a combination of a thermal resistor (for heat) and a ^{252}Cf external bombardment (to simulate the radiation environment for the materials). At the end of the project, we built and tested a working prototype.

FY 2006 Accomplishments

We assembled a second prototype with a power level of 338 µW output in the form of a 40 mV/16 mA source. This new prototype was accurately predicted by an improved linked thermal/electrical model that took into account both Peltier and Seebeck effects for the materials.

We fabricated the material rings using wire electron discharge machining (EDM), and demonstrated high yields for the devices with low electrical loss. We demonstrated a method for quadrupling the voltage by dicing the stack into four separate components and rewiring them in series, getting the voltage to a 160 mV loaded-output voltage. At this level, a specially designed bootstrapped voltage up-converter took this voltage to 2.6 V at 38 percent efficiency.

The final system volume was 4.3 cc and demonstrated a power density of 80 µW/cc, which is the smallest, most efficient (for its size) thermoelectric source ever built. Previous systems demonstrated a maximum of only 60 µW/cc in a system volume of 10 cc.

Significance

This project provides the potential of a long-duration, small, continuous power source at usable voltage for several different applications. We laid the groundwork for developing this into an application and completed the testing to show performance over time. We estimate that with this project, small thermoelectric sources at Sandia went from a technology readiness level of about 2 to level 3.

Advanced Material Applications of Precision-Deposited and Free-Form-Fabricated Energetic Materials

79876

J. Cesarano III, A. S. Tappan, J. N. Stuecker

Project Purpose

The overall goal of this project is to couple free-form-fabrication/precision deposition with energetic materials for applications requiring irreversible changes in properties. We are developing a deposition technology that allows designers to put strategic materials in strategic locations of components and to perform an irreversible strategic function.

There are two types of material systems/applications of interest: 1) precision deposition of an energetic material that functions as an explosive to generate heat, light, high pressure, and so on, with the objective to energetically perform a function; and 2) deposition of materials that (when initiated) function as reactants for self-propagating high-temperature synthesis to produce a material property change and/or volume change to irreversibly effect the operation of a component.

FY 2006 Accomplishments

Materials

We concentrated on systems in esters, thus reducing reaction during sample preparation and allowing a wider variety of binder systems to be used. We developed dispersion formulations for aluminum and nickel as well as a complete thermite formulation using mixed esters as the dispersing medium. We performed product searches and consulted with other groups on desirable gasless binder systems. Through consultation, we identified two products that will be studied with our systems. We also acquired a dual planetary mixer, thus increasing the quality of our slurries and of our formulation safety.

We selected the energetic material system Ti/2B for several reasons. It is extremely exothermic, gasless, and the product, TiB_2, is sufficiently electrically conductive to serve multifunctional purposes. While this system is extremely energetic, it is difficult to ignite due to the high melting temperature of the reactants. We addressed this issue through selecting fine-particle reactants, only recently available, and also by adding material(s) to tune the ignition and propagation through more facile side reactions. These are both established techniques to tune the characteristics of energetic materials.

We conducted research into formulation optimization with respect to energetics as well as casting formulation. We developed an efficient formulation method in which small-scale quantities of multiple variations of a material system are optimized through calculations, then formulated and tested to achieve the desired energetic properties. We developed a first-generation castable slurry based on this material system and will continue to improve it as we optimize the formulation for its energetic and fired material properties.

Conformal Deposition

We developed a trunnion, with rotation and tilt axes that convert our robocasting platform into a five-axis conformal printer. In principle, the five axes of control allow maximum printing precision because the printing tip can always be normal to the printing surface. In practice this can only be achieved with very sophisticated software capable of generating printing paths with coordinated motion among five axes.

We incorporated the trunnion, developed the mathematical models for five-dimensional coordinated motion, and formalized the architectural design of the software. The state of development is such that conformal tip-normal printing can be completed on symmetrical nonplanar surfaces. We demonstrated tip-normal printing on a hemispherical surface. We formulated Mock 900-13, which is a viscosity mock for the high explosive XTX-8003, into a printable paste.

Significance

This project is relevant to national security, particularly to enhancing nuclear surety through the development of technologies that facilitate mission needs for future components. There is specific relevance for nuclear weapon components that can use precisely deposited materials with dynamic material transitions as part of their function.

Spin-offs pertinent to advanced production of neutron tubes, micropower systems, and novel catalysts will benefit multiple strategic goals.

Refereed Communications

J. Cesarano, P.G. Clem, J.N. Stuecker, and M.K. Niehaus, "Direct-Write Approaches to Fabrication and Precision Printing at Sandia National Laboratories," in *Proceedings of the IMAPS/ACerS International Conference and Exhibition on Ceramic Interconnect and Ceramic Microsystems Technologies*, pp. 1-4, April 2006.

J. Cesarano, P.G. Clem, J.N. Stuecker, and M.K. Niehaus, "Direct-Write Approaches to Fabrication and Precision Printing at Sandia National Laboratories," in *Proceedings of the IMAPS Conference on Ceramics Interconnect and Ceramic Microsystems Technologies*, April 2006.

Other Communications

A.S. Tappan, R.J. Pahl, A.M. Renlund, J.J. Nogan, W.C. Sweatt, and F.B. McCormick, "Optical Pyrometry Temperature Measurement of Miniature Charges of MIC Nanocomposite Thermite," in *Proceedings of the 33rd International Pyrotechnics Seminar*, July 2006.

Remote Sensing of End-Event Timing for High-Fidelity JTAs

93414

N. R. Hilton, J. M. Van Scyoc, K. H. Tieu, R. S. Tilley

Project Purpose

A key tool of stockpile evaluation is the flight test of Joint Test Assemblies (JTAs), which are stockpile weapons rebuilt without key nuclear materials and which usually have large amounts of instrumentation to gather, process, and transmit weapon component and system performance data. However, there are also uninstrumented JTAs, which are sometimes called high-fidelity JTAs or simply Hi-Fis, from which little or no component or system data are obtained.

Flight testing of Hi-Fis is viewed as desirable or necessary because they most closely represent war reserve (WR) hardware. Similarly, minimizing the use of JTA-specific hardware and gathering more information about each function from instrumented JTAs are desirable. Flight-test opportunities continue to decline because of limited assets and funding; therefore, it is imperative to gather all possible information from each JTA flown, including Hi-Fis.

Any instrumentation added to a Hi-Fi must minimally impact the WR configuration. As a result, it is desirable and often required that some signals be remotely sensed. The Category A, or "yield-at-target," measurements indicate the correct implosion, neutron generator, and boost-gas functions.

Methods for remote neutron monitoring have been developed in other projects, which when combined with a method of remotely sensing the timing of the detonation would comprise all the data necessary to score neutron generator performance in a Hi-Fi. The first purpose of this project is to develop such a method for remote sensing of end-event timing.

Although not a specific goal of this project, we discussed concepts of collecting data in a Hi-Fi for the other two crucial measurements (i.e., implosion timing and gas transfer). Collecting such data from Hi-Fi tests where we currently get nothing would be significant for Sandia's stockpile evaluation mission.

For instrumented JTAs, a key deviation from WR is the breaching of the exclusion region to instrument the firing-set output, which is generally accomplished by current viewing transformers (CVTs). In the WR stockpile, CVT connections across the exclusion region boundary are sealed off. In addition to the loss of WR fidelity by breaching the exclusion region, it is challenging to obtain CVT data in the presence of the electromagnetic interference caused by firing-set operation. Therefore, the second goal of this project is to develop a means for assessing firing-set output without electrical connection, which would address both hardware fidelity and signal-to-noise ratio issues.

FY 2006 Accomplishments

We assessed the viability of enhanced JTA end-event instrumentation, for both uninstrumented high-fidelity and instrumented configurations.

Major effort went into characterizing the electromagnetic interference (EMI) environments present in firing events, with experiments on various configurations. Initial tests on low-voltage capacitive-discharge units revealed that significant energy in the frequency range of interest, 50 kHz to 50 MHz, is broadcast, even from low-current discharges. As expected, subsequent testing with different WR components in various configurations demonstrated that the remote detection of the higher currents in actual explosive firing events is strongly affected by the shielding effects of metal casings.

Testing of a JTA terminal data analyzer, now in development, also verified the presence of significant fire-down EMI signatures, if sensing elements are in proximity to the firing set and/or detonators.

In other tests, we collected large-amplitude EMI signals using wide-band antennae placed a few feet from detonator simulators, explosive valve actuators, and MK4A/W76-1/JTA1 firing sets and other WR components in a lab bench configuration. In the latter, all WR explosive components were enclosed in a blast containment chamber, or "boom box," but the EMI signal levels were still very high. However, in a full JTA1 ground test, where our instrumentation was completely external to a sealed reentry body, we determined that reliably capturing end-event EMI signals requires the sensor elements to be less shielded from the firing set.

For the wireless CVT portion of the project, we determined that the original architecture of a signal-sourced power supply and data acquisition system is infeasible. In that architecture, the signal to be measured passes through a passive delay line, by which time the power supply is stable. The delay line timescale is consistent with the timing of the shock environment; however, it is not possible to design a power supply with the required settling time over all ranges of firing-set output currents. Therefore, we conceived of two alternate architectures.

In one approach, an optical CVT relies on Faraday polarization rotation in an optical fiber coiled around the firing-set output cable. The main telemetry module monitors the polarization shift via crossed-polarizer extinction. Initial calculations showed this approach to be sensitive over the range of firing-set output currents.

The second approach uses the frequency shift of an oscillator circuit under an applied magnetic field. The underlying signal both provides power and modulates the natural frequency of the circuit. The frequency is tuned to a band where the EMI background is low; therefore, the resultant frequency-modulated CVT signal can be discriminated by an RF (radio frequency) receiver in the main telemetry module. In this approach, the transmitted signal amplitude can vary by several orders of magnitude and still be monitored.

Significance

This project directly ties to the National Nuclear Security Administration mission of certifying safety and reliability of deployed nuclear weapons. One goal of this project is to demonstrate some of the technical means needed to obtain scoring data from otherwise unscored Hi-Fis. As JTA flight opportunities decrease, the need for obtaining more and higher-fidelity information from every asset consumed is critical.

Another goal is to show that a higher-fidelity configuration for instrumented JTAs is possible without breaching exclusion regions while still providing detailed firing system data. The successful conclusion of this project will provide a new set of advanced JTA tools that are too risky to develop under ongoing JTA programs. It will also contribute to the Integrated Stockpile Evaluation goal of obtaining more data from fewer stockpile return units.

A Modern Nuclear Weapon Communications Architecture

93415

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Project Purpose

The purpose of this project is to develop a modern, plug-and-play weapon bus kernel that will provide a common backbone for future warhead designs. This backbone would allow the system to interface with legacy systems, such as existing weapon carrier platforms, or newly designed host platforms, such as the System 2 interface. It will also increase reliability by reducing cable interconnections, facilitate reconfigurability to achieve new capabilities, and lower cost to maintain and conduct surveillance by allowing plug-and-play capability for sensors and components.

We will conduct a study to identify the system and subsystem level needs and work with the system, subsystem, and nuclear safety groups to identify all the issues. The study will produce:

- A list of basic attributes describing features that a communications bus needs to provide to support system, subsystem, and safety needs. This will guide the protocol study.
- Documentation giving nuclear safety guidance using an internal weapons bus. This will determine what features are allowed and what features would cause issues with using a given communications protocol.

We will conduct another study using the information uncovered in the needs/safety study. The main purpose of the protocol survey study will be to determine if any of the existing serial commercial communications protocols used by industry can support our special needs.

We will study possible physical-layer architectures to identify candidate layers that will provide a robust error-free operation in our harsh environments. We will investigate methods for providing fault recovery at the physical layer level as well as at the logical level (within the communications protocol and node logic). We will explore both electrical- and optical-based physical layers and identify the maximum bus length and operating environments to support all of our possible uses.

We will study embedded monitoring systems and features to include in the communications protocol that support embedded monitoring. In order to achieve transformation, future systems will need to include more built-in testability features to support built-in testing during all phases of the stock pile lifetime and reduce the number and complexity of external testers.

We will address issues associated with powering up the bus to communicate with the embedded monitoring functions. We will also investigate testability. Any candidate communications protocol used in a future system should include features that would assist in the testing effort.

Existing systems are designed in a way that requires several versions of a tester to be developed for use at the different facilities where the testing is performed as the system progresses through its lifetime (development, production, flight testing, and stockpile monitoring). A standard communications protocol would make possible a common core tester design that can be used during each phase of testing. This would include all locations across different programs within the weapon complex, resulting in transformation of the way testing is done.

FY 2006 Accomplishments

Working with the nuclear safety department, we conducted a detailed study to identify and address nuclear safety issues relating to an internal nuclear weapon communications bus. The study outcome is a set of design guidelines that will be used for the selection or development of a nuclear weapons bus communications protocol and is documented in memo format. In addition, we identified basic system and subsystem needs.

We also conducted a study of existing communications protocols used by industry and aerospace (automotive, IC industry, avionics, military, and space agencies) to determine if any of the standard protocols met our special needs. We determined that all of our critical attributes could not be meet by any single protocol.

Some of our key attributes were:

- Security/Safety
- Testability
- Reliability
- Radiation Hardness
- Flexibility
- Simplicity
- Power Management

The standard industry protocols we studied include:

- Flexray – Automotive industries' drive-by-wire and break-by-wire communications protocol
- CAN – (controller area network) automotive industry bus protocol
- I squared C – IC industry inter chip serial communications protocol
- Spacewire – European Space Agency serial communications protocol
- 1553 – military serial communications bus protocol
- MOST – automotive multimedia protocol
- AFDX – version of ethernet used by the avionics industry
- USB – computer industry serial protocol
- Ethernet – computer industry network protocol
- Firewire – computer industry high speed serial protocol

We also looked at Byteflight and TTP/C, which have been replaced by Flexray. Other protocols, RS232, for example, were quickly eliminated from the protocol study and are not included in the list.

We determined that a new communications protocol based on the best attributes found in industry standard protocols needs to be developed for nuclear weapon use. We made significant progress in defining the new nuclear weapon communications protocol, called "Foundation," and consulted nuclear safety, systems, and subsystems groups during its development.

We are also developing a Java-based model that uses the Sandia-developed simulation environment, called "Orchestra," to study system, subsystem, and nuclear safety needs. In addition, the functionality of the new communications protocol (at a systems level) will be verified by this model, which simulates Foundation at the bit level and represents real timing.

For the physical layer, we are considering both optical and electrical options. Sandia VCSEL (vertical-cavity surface-emitting laser) optical technology is being investigated as a possible physical layer for use in a weapon communications bus. We generated a paper design for a VCSEL-based prototype optical physical layer that will be built and tested in FY 2007. We also developed a paper design for an electrical physical layer that will be built and tested in FY 2007.

We started development of the embedded analog monitoring concepts that, when connected to the bus, will provide built-in testability features.

Significance

Some of the key R&D accomplishments will impact future systems in the following areas.

Testability

Reliance on external custom testers for each weapon system could be reduced. Features developed and placed in the new communications protocol will now allow future systems to include built-in testing and monitoring. The possibility of a common tester that could be used across mutable systems is now possible, thus reducing system cost and allowing design reuse.

As future systems are being developed, the ease of acquiring additional data during development is greatly enhanced as all traffic on the bus would be captured by the bus monitor. The ability to acquire more data during development will allow better characterization of system performance. In addition, as a system begins to age, system performance can be monitored with better fidelity to look for early indications of possible problems long before system performance is impacted.

Features placed into the communications protocol will allow future bus-based testers to capture every packet sent on the internal bus for generating a complete time history of all activity. This opens the possibility of more detailed system analysis during development and stockpile monitoring activities using automated processes. Attributes that define the functionality of a bus monitor were identified in FY 2006. A version of the bus monitor will be developed in the simulation environment that will allow the analysis software to be tested and debugged before real hardware is available.

A bus-based protocol allows the possibility of stand-alone testing of subsystem modules without other subsystem nodules being present. Messages emulating the functionality of the other modules would be computer generated and inserted into the system. This would result in the transformation of the subsystem development process for future systems. The ability to test subsystem modules during development with computer-generated stimulus reduces the need for additional hardware during development testing.

Features in the communications protocol would allow the possibility of reconfigurable built-in JTA in future systems. Future JTA systems would have full access to all data on the bus and could be configured to acquire different data on each flight test. JTA data lists could become dynamic based on the phase of the flight, thus making better use of the JTA bandwidth.

Flexibility

The new communications protocol provides plug-and-play capability and the possibility to reconfigure systems as mission needs change. Features that are no longer required can be easily removed.

The use of a common communications protocol opens the possibility of design reuse of functional elements between systems. This could provide opportunities for reduced cost and development time on future systems, taking advantage of already qualified functions from other designs.

Nuclear Safety

As a result of the nuclear safety study, undesired features found in most commercial communications protocols were excluded from the new communications protocol. In addition, some new features that enhance safety were identified and added to the protocol.

Improved Power Source for Doubling the Exchange Time Interval of LLC

93416
G. Nagasubramanian, K. R. Zavadil

Project Purpose

The purpose of this work is to understand the depression of the operating voltage of the carbon monofluoride $(CF_x)_n$ chemistry and to offer solutions to increasing the operating voltage. Increasing the operating voltage would increase the delivered energy to potentially double the exchange time interval of limited life components (LLCs).

This chemistry has the highest theoretical specific energy, at 2200 Wh/kg, of any lithium primary battery known. In spite of this, the delivered energy is, at best, 30 percent of theoretical. The main cause of the reduction in the delivered energy is the depression in the operating voltage. The operating voltage is approximately 2.5 V, which is ~ 0.8 V lower than the open circuit voltage.

A lot of unsubstantiated theories were published describing possible causes for the voltage reduction. We took up the challenge to decipher the reaction mechanism and to ultimately offer a solution that would improve the operating cell voltage to > 2.5 V. This LDRD work is both fundamental and applied in nature.

The fundamental aspect of this work focuses on understanding the kinetic limitations of the discharge reaction of the $Li/(CF_x)_n$ cell chemistry using a suite of modern surface analytical tools such as scanning tunneling microscopy (STM), atomic force microscopy, and time-of-flight secondary ion mass spectrometry (TOF-SIMS), as well as performing in situ x-ray analysis while discharging pouch cell to identify intermediate species.

The applied aspect of this work focuses on selecting the best-performing material for the fundamental study. This work involves preparing good quality electrodes that exhibit reproducible behavior.

FY 2006 Accomplishments

Our important FY 2006 accomplishments:

- We prepared highly reproducible $(CF_x)_n$ electrodes.
- We optimized pouch cell configuration to maximize x-ray signal from the active material.
- We performed in situ x-ray studies while the $Li/(CF_x)_n$ cell was being discharged.
- We demonstrated, using STM, that individual $(CF_x)_n$ particles sustain tunneling current.
- We demonstrated ex situ compositional characterization of particles using TOF-SIMS.

We found a way to anchor particles on gold by relying on the shape of these particles and van der Waals attractive forces between the particle and an alkylthiol-terminated gold surface. These particles can be observed in atomic force micrographs. Particles ranging from tens of nanometers up to microns appear stable. These same particles can be imaged using STM, so finite electrical conductivity exists to support tunneling between the particles and the tip. Particle orientation allows for structural mapping along the c-axis, or basal plane, and the a-axis, or graphene plane, terminus.

The in situ x-ray measurement did not identify any intermediate species in the discharge reaction. There are various possible reasons the intermediate was not detected, including:

- the intermediate is amorphous
- the intermediate is present in extremely small amounts
- the x-ray technique was too slow to detect the intermediate.

Significance

To our knowledge, Sandia is the first in the world to coat $(CF_x)_n$ electrodes with polyvinylidene difluoride (PVDF) as the electrode binder. This accomplishment is a significant one to the general science and technology community because it allows fabrication of cells with high capacity. This capability is being integrated in to the Advanced Systems and Technologies program, where we are making large capacity Li/$(CF_x)_n$ cells.

On the fundamental aspect of this project, we focused our preliminary activities on developing the capability to conduct isolated cathodic discharge and characterization on single particles of carbon monofluoride. Our initial efforts were focused on fixing single particles to suitable substrates and demonstrating the probing of these particles. We emphasized scanning probe microscopy, initially as an ex situ probe, but with the intent of conducting in situ imaging during local cathodic discharge.

We conducted a preliminary demonstration of single-particle compositional characterization using TOF-SIMS. Particles were fixed to amorphous carbon TEM grids for analytical electron microscopy and diffraction on individual particles. If we crack the reaction mechanism and pinpoint the rate-limiting step, we will have the potential to develop a battery that has double the energy of the current Li primary chemistry (Li/($SOCl_2$) being used in nuclear weapon applications.

Increasing the Accuracy of Vision-Based Dimensional Metrology

93418
H. D. Tran, M. G. Hankins, B. Jokiel Jr., K. M. Shilling, A. A. Claudet, A. D. Oliver

Project Purpose

The purpose of this project is to develop a way to calibrate vision-based dimensional inspection equipment, which would improve the accuracy of existing equipment. An additional objective is to develop methods to calibrate and verify the performance of microcoordinate measuring machines (microCMMs).

The major contributor to measurement uncertainty of vision-based, dimensional inspection equipment is the artifact – typically a grid plate – that is used to calibrate the vision-based system. A grid plate is a glass plate deposited with precision cross hairs. The relative locations of the cross hairs are calibrated and used to calibrate the vision-based machine. A fundamental problem is that it is a vision-based system being used to provide the master calibration for a grid plate. To get better accuracy, a better master with a better vision probing system is needed.

The project focuses on alternate calibration artifacts. High-precision CMMs are capable of achieving accuracies that are as good as, or better than, the resolution of vision-based dimensional inspection equipment. We, therefore, designed a calibration artifact with geometries that are compatible with vision-based probing (for vision systems) and touch probing (for CMMs). We call this artifact a hybrid because it can be measured using more than one measurement technology.

By using a slow, but extremely accurate, touch probe CMM to evaluate the geometry of the hybrid measurement artifact, we can provide a very accurate calibration of its geometry. We can then use this artifact to calibrate vision-based dimensional inspection systems, where the limiting factor in accuracy was the accuracy of the grid plate.

FY 2006 Accomplishments

We resolved several potential obstacles. We found that commercial vision-based dimensional equipment can accept external error maps. We identified one additional potential obstacle: risks in using reflected illumination.

We investigated the possibility of using heterogeneous material fabrications and alternate traceability to develop an intrinsic dimensional standard at micrometer length scales, and demonstrated that this was not practical.

We completed our first design of a family of structures for two-dimensional calibration, which also includes one structure for three-dimensional calibration. This design has been built and is being evaluated.

We demonstrated the feasibility of imaging bulk micromachined silicon at high resolutions on vision-based equipment, but we have not yet quantified optimal illumination conditions. Preliminary tests indicate that the edge determination of the bulk micromachined silicon artifact is insensitive to illumination intensity, which is a good result.

We met with dimensional metrology personnel at two other National Nuclear Security Administration facilities (Lawrence Livermore National Laboratory and the Kansas City Plant), and they have agreed to participate in an inter-laboratory comparison with us, based on our improved calibration artifact.

We obtained microfabricated touch probes from Physikalisch-Technische Bundesanstalt (PTB, the German national metrology institute) that are capable of submillinewton probing forces.

Our analysis of measurement uncertainties associated with the repeatability of the Moore coordinate CMM, together with expected errors in flatness and surface finish from the bulk micromachining of silicon, combine to produce measurement uncertainties on the order of 30 nm (at a 95 percent confidence level).

We filed two invention disclosures, had one paper accepted at the American Society of Mechanical Engineers' 2006 International Conference on Manufacturing Science and Engineering, and had another paper accepted for the American Society for Precision Engineering 2006 Annual Meeting.

Significance

Better accuracy and faster measurements are always desirable. Sandia's Primary Standards Lab, which is accredited by the National Voluntary Laboratory Accreditation Program, is the key facility for ensuring measurement integrity throughout the nuclear weapons complex. This project addresses a key need for future dimensional metrology: metrology at mesoscale for miniaturized parts and assemblies. Mesoscale is defined here as ranging from a few micrometers to approximately one hundred millimeters.

Stockpile extension programs and other new designs are increasingly emphasizing miniaturization, especially for electromechanical components at mesoscale (mm to sub-mm). These include some of the key components in nuclear weapons.

Measurement of mesoscale components is a challenging problem. A successful metrology program will ensure the quality of electromechanical components fabricated using microelectromechanical systems and LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) techniques, or even with other production methods such as precision machining or laser-engineered-net-shaping, and help not only to reduce costs, but also to assure future stockpile integrity.

The three groups that comprise this research collaboration are metrology, advanced manufacturing, and microsystems packaging. This collaboration will enhance future rapid product development and deployment at Sandia. Successful production of these mesoscale artifacts will enhance not only Sandia's production metrology capabilities, but also production dimensional inspection across the DOE complex.

There is potential interaction with industry and the National Institute of Standards and Technology to disseminate the technology across a broader market, as the problem being addressed is a general measurement and manufacturing problem, and not tied specifically to stockpile components.

Refereed Communications

M. Shilling, A.A. Claudet, A.D. Oliver, and H.D. Tran, "Uncertainty Analysis for a Silicon Bulk Micromachined Dimensional Metrology Artifact," to be published in *Proceedings American Society for Precision Engineering (ASPE) Annual Conference*, Monterey, CA, October 2006.

A.D. Oliver, A.A. Claudet, and H.D. Tran, "Design of a Silicon Micromachined Artifact for Hybrid Dimensional Measurement," to be published in *Proceedings ASME Manufacturing Science & Engineering Conference (MSEC) 2006,* Ypsilanti, MI, October 2006.

High Kinetic Energy Ion Source

93421
J. M. Elizondo-Decanini, B. H. Cole, J. P. Brainard

Project Purpose

The purpose of this project is to demonstrate an ion source concept that takes advantage of small size and generates high-current densities and high local magnetic fields to produce high initial kinetic energy ions capable of overcoming the space-charge limitations of a typical ion source. Ion sources producing high initial kinetic energy ions can overcome, by several times, the Child-Langmuir space-charge limited current values given by the baseline equation with a $V^{3/2}$ dependence.

The baseline equation is obtained with the assumption that the initial ion kinetic energy is approximately 0. The ratio of this current to the one obtained with non-zero kinetic energy ions yields a maximum gain of as much as 8 times in current, depending on the ion initial kinetic energy. This ratio is for currents in the accelerating gap, not on the source output gap; this is a significant increase, and we want to demonstrate and use it to our advantage. However, even a theoretical ratio of 2 has been difficult to produce in a practical device.

The purpose of this project is to demonstrate that the theoretical higher current values can be obtained by using to our advantage what is otherwise considered a limitation.

FY 2006 Accomplishments

We completed all ion source models and preliminary calculations and the design for the initial test coaxial ion source. We fabricated three sources and are ready to test them. We completed the experimental setup, and the tests are in progress to obtain a reference value of current and ion energy using presently available ion sources. We will use the data from this test as a reference to benchmark the proposed ion source.

The experimental setup includes a laser heterodyne interferometer to measure plasma number density, a field-free long drift chamber to measure velocity distribution and charge state densities, and an ion energy analyzer to measure energy distribution. The experimental setup permits us to research and determine the specific features that affect the predicted theoretical behavior of high initial kinetic energy ions.

Significance

If the research produces the expected results (predicted from the theoretical analysis and models), ion sources capable of high initial kinetic energy will reduce the complexity and/or the number of components of a number of mission-critical components. Increasing the ion current capacity at constant input current, or reducing the input current at constant ion current, will benefit other applications, such as radiography, and result in:

- increased capacity
- larger margins
- higher reliability
- reduced input parameter requirements
- smaller size
- reduced number of components.

Advanced Manufacturing

Rapid Prototyping to the Nanometer Scale

67004

D. P. Adams, M. B. Sinclair, M. J. Vasile, T. M. Mayer, W. C. Sweatt, G. R. Burns

Project Purpose

The purpose of this project is to research new techniques for prototyping nanometer-scale components and devices. In particular, we are developing focused ion beam-based methods that precisely tailor feature dimensions and net feature shape.

We are investigating three novel in situ metrology techniques for real-time monitoring and feedback control of feature depth and shape during focused ion beam (FIB) processing: Michelson interferometry, secondary electron (SE) profilometry, and endpointing during single-point nanohole drilling. To our knowledge, this is the first attempt to combine real-time interferometry with focused ion beam methods for improved control of feature shape.

Secondary electron profilometry during ion exposure is an altogether new technique that has not been researched previously. This particular technique potentially offers submicron precision for metrology. Endpointing during nanohole drilling provides a method for controlled fabrication of small pores while avoiding large area, beam raster, hole-closure methods that may otherwise damage neighboring volumes of a device.

In order to take full advantage of various in situ metrology techniques, we are also researching numerous focused ion-solid interactions critical to nanometer scale device and component fabrication, including:

- studies of surface morphology evolution during bombardment
- atomistic processes that lead to morphology changes
- effects of morphology on yield (i.e., material removal rate)
- variations of morphology over a milled curvilinear feature
- the angular distribution of secondary electron emission for different materials (important for SE profilometry)
- effects of charge distribution/surface potential resulting from ion implantation.

FY 2006 Accomplishments

We developed and tested a novel, custom-built interferometer microscope for in situ monitoring of FIB-sculpted features. This instrument should enhance FIB milling as a tool for prototyping activities. The instrument operates within the target chamber of an FIB system (maintained at high vacuum) and noninvasively probes feature depth and shape.

We completed tests to evaluate the resolution of the interferometer, both in-plane and out-of-plane. The custom-designed optics offer 1.0 µm in-plane resolution and better than 5 nm out-of-plane resolution when operating at approximately 530 nm.

We analyzed how resolution is affected by the diameter of a pinhole contained within the center of a mirror and positioned just below the FIB exit aperture. This pinhole allows for simultaneous passage of an ion beam to the specimen surface during metrology.

We determined that the maximum slope (of a curved feature) measured by the custom interferometer is approximately 20 degrees. Additional optimization has focused on minimizing vibrational effects on resolution, since the interferometer is decoupled from the specimen fixture. We submitted a patent application on this method.

In addition, we completed the software that determines the required ion beam pixel dwell times needed to generate desired custom topographies. The software was developed in Visual Basic for operation with an FEI Company Magnum FIB column and its stream file-based user interface, and offers a numerical method to generating tailored surface topographies.

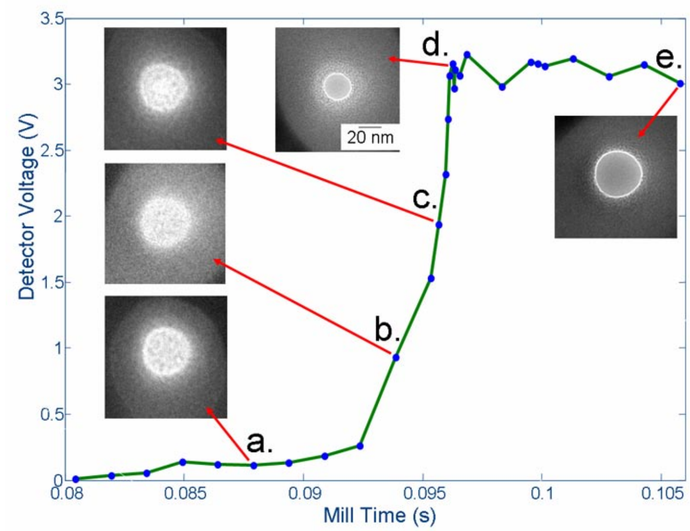
In a second approach, we researched an altogether new metrology technique, SE profilometry, for use during focused ion beam processing. This technique allows us to monitor feature shape – and potentially depth – during focused ion milling with improved (submicron) precision. We fabricated, installed, and tested a custom quadrant channelplate detector, amplifier electronics, and software. We determined the detector response for sloped target surfaces through experiment and compared it with earlier Monte Carlo simulations.

We investigated a third method for precise nanohole drilling. Direct ion drilling that avoids beam raster/hole closure methods is attractive because we avoid ion exposure of neighboring regions that might include a device. We developed methods that rely on a backside, high-energy channelplate detector for monitoring current in order to tailor feature size. We studied the detailed response provided by a backside channelplate detector about the onset of breakthrough, related this to various processes including forward sputtering and transmission, and demonstrated how this can be used for endpointing.

Transmission electron microscopy of drilled specimens shows that this method can be used to reproducibly define the exit-side-hole diameter equal to 20 nm when drilling through 200 nm-thick silicon nitride membranes. We further detailed the hole morphology (net shape, height of the boundary with respect to the original surface).

Significance

Our research should greatly enhance the capability of FIB methods for nano- and microdevice prototyping and failure analyses methods. These activities are currently of great interest to the R&D community, industry, and national security missions.



Results from single pixel focused ion beam drilling showing backside detector response as a function of mill time. Transmission electron microscope image d. shows evidence of breakthrough and a 20 nm diameter hole.

The techniques involving interferometry and SE profilometry should find use in device prototyping, reverse engineering, and failure analyses.

FIB sputter fabrication methods can be used to pattern almost any solid material. After completion of this project, FIB methods will offer feature-shape control, some capability for controllably defining features to 10 nm dimension, and the ability to endpoint those features.

An additional metrology technique we investigated should find near-term interest in the R&D community. The technique involves precise drilling of 10-100 nm diameter "through holes" in membranes while endpointing and includes monitoring breakthrough and preceding events. A great deal of interest in this idea has surfaced over the past year from researchers involved in desalination/water purification studies and researchers investigating translocation of biomolecules (e.g., deoxyribonucleic acid) through mesoporous membranes. Our methods now permit us to repeatedly tailor membrane hole diameter from 10 to 100 nm.

Refereed Communications

D.P. Adams and M.J. Vasile, "Accurate Focused Ion Beam Sculpting of Silicon Using a Variable Pixel Dwell Time Approach," *Journal of Vacuum Science and Technology B*, vol. 24, pp. 836-844, 2006.

D.P. Adams, M.J. Vasile, and T.M. Mayer, "Focused Ion Beam Sculpting Curved Shape Cavities in Crystalline and Amorphous Targets," *Journal of Vacuum Science and Technology B*, vol. 24, pp. 1766-1775, 2006.

D.P. Adams, T.M. Mayer, M.J. Vasile, and K.A. Archuleta, "Effects of Evolving Surface Morphology on Yield During Focused Ion Beam Sputtering of Carbon," *Applied Surface Science*, vol. 252, pp. 2432-2444, 2006.

Other Communications

K.A. Archuleta, J. Fenton, M.J. Vasile, S. Campin, D.P. Adams, and J.E. Fulghum, "Scanning Probe Microscopy and X-Ray Photoelectron Spectroscopy Investigations of Focused Ion Beam-Irradiated Samples," presented at the 52nd AVS International Symposium, Boston, MA, November 2005.

D.P. Adams, M.B. Sinclair, T.M. Mayer, M.J. Vasile, and W.C. Sweatt, "Optical Interferometer Microscope for Monitoring and Control of Focused Ion Beam Processes," in *Proceedings of the Microscopy and Microanalysis 2006*, p. 1280CD, August 2006.
D.P. Adams, "*Focused Ion Beam Shaping*," presented (invited) at Harvard University, Cambridge, MA, March 2006.

D.P. Adams and M.J. Vasile, "Accurate Focused Ion Beam Sculpting of Curved-Shaped Features in Metals and Amorphizable Solids," presented at the 52nd AVS International Symposium, Boston, MA, November 2005.

M.J. Vasile, T.M. Mayer, D.P. Adams, M.B. Sinclair, and W.C. Sweatt, "Optical Interferometric Microscope for Real-Time Monitoring and Control of Focused Ion Beam Processes," presented at the 52nd AVS International Symposium, Boston, MA, November 2005.

Robust Manufacturing of Gel-Based Components for a Wide-Array of Applications

67005

P. J. Cole, R. V. Baron, D. R. Tallant, J. L. Lenhart

Project Purpose

The development of nonaqueous polymer gels is important to Sandia because the gels enable key electrical components in ongoing life extension programs (LEPs). These electrical components represent a critical technology for Sandia, both now and into the future, as they are the only available components that provide a unique combination of properties in conjunction with nuclear safety features.

The gels initially developed for this application were nonaqueous gels consisting of a crosslinked polymer swollen with a high-dielectric constant solvent. Poor polymer-solvent phase behavior has been tied to the inferior mechanical properties that lead to component failures. Based on gel structure, the most influential issue impacting the material properties was polymer-solvent phase behavior. While phase behavior is largely determined by thermodynamics, it is also affected by the method used to manufacture the gel-based components.

A primary purpose of this project was to establish a relationship between the gel fabrication procedure and the resulting mechanical properties, gel microstructure, and phase behavior. We showed that the preparation method dramatically altered the final mechanical properties of the gel. Specifically, the preparation method used to manufacture gels comprised of solvent-swollen crosslinked networks impacts the final properties through two screening mechanisms. Exploiting these screening mechanisms enables various gel properties to be tuned somewhat independently and tailored to an application.

Through establishing this relationship, this project provided not only the critical scientific foundation for ensuring successful implementation of gel-based components in nuclear weapons, but also for exploiting gels of various chemistries in a variety of other applications.

FY 2006 Accomplishments

We determined that phase separation can be used to control properties. We showed that the addition of solvent can suppress the plateau modulus (effectively gel stiffness). Furthermore, the gel glass transition temperature (Tg) is suppressed until the solvent phase separates from the gel. Further addition of solvent does not lead to a change in the Tg, it only creates larger pockets of solvent. With this knowledge, the plateau modulus and Tg can be tuned independently by controlling the phase separation temperature.

The presence of solvent during cure changes the gel entanglement density. Through comparison of three types of gels, we showed that the fabrication methodology can dramatically impact gel properties. Gels that were made by curing the polymer network and then adding solvent have moduli that depend only slightly on the solvent content (less than a linear dependence). Conversely, gels in which the crosslinked network is formed in the presence of solvent show a strong dependence on the solvent content (more than a squared dependence). This demonstrates our ability to control the number of entanglements in the gel network and, similarly, the “openness” of the gel microstructure.

Swelling of networks formed in the presence of solvent is dramatically altered, providing evidence of microstructural changes in the gel. Network swelling of crosslinked polymers represents a thermodynamic balance between chain stretching and beneficial enthalpic contributions from interactions of a good solvent. More open network microstructures are formed by creating the gel network in the presence of a good solvent. Removing the original solvent through an extraction process leaves a network that can be dramatically swollen compared to the dense networks cured without solvent. Our experimental results demonstrate factors of 3-5 enhancement in swelling behavior.

Varying the network chemistry stabilizes the microstructure, making gel technology more broadly applicable. We demonstrated the processing and phase behavior impact on microstructure for three dramatically different gel chemistries. The implication is that the microstructural control can be applied to systems such as water-based gels or rigid networks that might be useful for molecular templating.

Solvent molecular weight changes impact gel response time and mechanical/adhesive properties. We explored the impact of solvent molecular weight by comparing silicone gel systems in which the solvent molecular weight was tens of thousands of grams per mole to systems in which the solvent was a small molecule on the order of 100 grams per mole.

We showed that transport of the solvent through the gel can be increased significantly as the molecular weight increases, leading to stress relaxation and energy dissipation. This further expands the application of this work, as well as gel technologies. This also provides a framework for understanding the slow migration of high molecular weight solvent (or sol) that presents an aging mechanism for gels in weapons applications.

Significance

We had significant success in bringing together the various aspects of the project to expand the applicability of gel technology both internal and external to Sandia. Our development of the links between gel chemistry, processing methodology, and resultant microstructure and properties expanded the application base by demonstrating how thermal transitions, mechanical properties, adhesive properties, and microstructure can be tuned to a wide-variety of specifications.

By looking at a variety of gel chemistries and extracting the processing solvent, we showed that microporous structures can be generated. Similarly, using the same strategy with a templating molecule, we demonstrated the possibility of achieving enhanced selectivity, which may be particularly relevant for sensor applications.

Selecting a templating molecule for a particular chemical, biological, or radiological species provides the opportunity to develop sensor systems for each of these threats. Perhaps most important is the science-based understanding we developed of the gel systems most relevant to weapons systems. This knowledge base will allow weapons designers to use gel technology with confidence in future weapons or LEPs.

Refereed Publications

J.L. Lenhart, P.J. Cole, B. Unal, and R. Hedden, "Non-Aqueous Polymer Gels," to be published in *ACS Symposium on Polymeric Materials for Anti-Terrorism and Homeland Defense*.

J.L. Lenhart and P.J. Cole, "Adhesion in Soft, Solvent-Swollen Polymer Gels," to be published in *Journal of Adhesion*.

Other Communications

J.L. Lenhart and P.J. Cole, "Soft Polymeric Materials: Fundamental Understanding to Practical Application," presented (invited) at University of Louisville, Louisville, KY, 2006.

J.L. Lenhart and P.J. Cole, "Materials Research at Sandia: Applied Science and Engineering," presented (invited) at University of Connecticut, 2005.

J.L. Lenhart and P.J. Cole, "Soft Polymeric Materials: Fundamental Understanding to Practical Application," presented (invited) at Drexel University, Philadelphia, PA, 2006.

Injection Molding of Net-Shape Active Ceramic Components

67007

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Project Purpose

The purpose of this project is to develop an injection molding process for fabricating lead-based ferroelectric ceramic components used in current weapon systems.

The approach will significantly reduce pressing- or machining-induced defects. Ceramics with these defects are more susceptible to handling damage or high-voltage breakdown in subsequent processes. For example, more than 20 percent of ceramic components are lost due to cracking and chipping in our manufacturing process. More importantly, by eliminating machining processes, this net-shape forming approach can save 18 percent of the touch labor cost, and significantly minimize the amount of hazardous-waste stream in our production line.

We concentrated our effort on (1) optimizing the injection molding process, (2) evaluating manufacturability, (3) minimizing lead loss during the high-temperature sintering process, and (4) continuing to improve rheology and computational modeling.

Fabrication of high-quality injection-molded ceramic components requires a basic understanding of the interrelationships between the powder characteristics, the thermal-rheological control of the molding material, the pressure of material in the mold cavity, and tooling parameters. Based on results from last year's experiments, we refined our injection molding parameters to eliminate the cavity formation.

We performed manufacturability studies to determine repeatability and reproducibility of the whole injection molding process from initial raw material preparation to final dimensional control of sintered parts. Finally, we determined the capability of this process based on what specifications would yield a process capability index (C_{pk}), of 2 (parts per million defective) for the various measurements.

The objective of these evaluations was to demonstrate that injection molding can meet all the stringent requirements of weapon components.

The manufacturing of lead-based electroceramics with reproducible performance can be complicated because of lead loss associated with the volatility of lead oxide at the temperatures used to sinter these materials. To help compensate for lead loss during calcination and sintering, excess lead can be added to the batch formulation. In addition, lead-containing burial powders can be used to help control lead loss during sintering by equilibrating the lead activity between the ceramic compacts and the atmosphere.

We experimentally developed a new technique that combines the balance between the amount of sample and burial powder used in the sintering process and the application of setter tiles to minimize thermal diffusion at high temperatures to suppress the lead loss in ceramic components. This process renders a unique near-shape forming capability for sintering high-volatility lead-based electroceramics where additional machining processes to remove a lead deficient surface layer can be eliminated.

We used computational modeling and rheology studies to help design and troubleshoot the injection molding process. We obtained flow solutions using a fully-coupled, finite-element based, Newton-Raphson numerical method. We solved the evolution of the free surface with an advanced level set algorithm. This approach incorporates novel methods for representing surface tension and wetting forces that affect the evolution of the free surface.

FY 2006 Accomplishments

We completed a preliminary process capability analysis for the injection molding of lead-based ceramic components. We determined the upper and lower limits of the physical dimension variation with a C_{pk} of 2 along three axes.

We established a linear correlation between the amount of variation and the physical dimension of the part, which permits a first-order estimation of whether a component fabricated by this technique can meet product specifications (i.e., tolerance) when the design is changed.

In addition, results show that there are operator-to-operator differences in the compounding process that lead to small, but statistically different, width uniformities. This observation suggests that subtleties in the compounding process are important and need to be refined before a more robust process is established.

We significantly reduced the amount of weight loss during sintering using a traditional double inverted crucible technique with a balance of weight between samples and buried powder per crucible volume. Based on three experimental lots (approximately 200 parts/lot), the average weight loss was 0.18 percent (<0.50 percent is considered to be a good process), indicating that the process was effective to suppress lead loss.

We compared Raman spectra from the surface and the interior of a sintered specimen, and performed a multivariate, chemometric analysis on these spectra. The discrimination analyses showed that there appeared to be some depletion of lead in the surface area relative to the interior material, but we estimated the relative lead depletion to be much less than 0.1 percent of its average concentration. These results suggested that there was almost no “skin” effective on the surface of these parts; therefore, no additional machining was required, indicating that this process will miminize the amount of hazardous waste stream.

This year we completed two-dimensional (2D) and fully three-dimensional (3D) models. The 2D models used complex rheology developed from capillary viscometry measurements as functions of time, temperature, and shear rate. We ran isothermal and nonisothermal simulations using a highly shear-thinning Carreau fluid model with a Williams-Landel-Ferry (WLF) temperature dependence.

Isothermal results showed that the viscosity at the walls and in the gate was shear-thinned to 400 poise, whereas the center of the channel and the free surface had a viscosity two orders of magnitude higher. The nonisothermal simulations showed that the hot fluid cools to the mold temperature within three channel diameters of the inflow, with the downstream results looking more like the isothermal cases.

In the 3D simulations, which were limited to Newtonian fluids, we investigated several distributor designs and orientations. We also performed laboratory experiments in a transparent mold to validate these simulations. Results showed that the original vertical orientation of the mold left voids at all four corners of the mold on the side opposite the distributor inflow.

In contrast, a horizontal orientation (with the vent moved to the top, the side opposite the distributor inflow) showed bubbles trapped only on the top two corners nearest the inflow. Simulations showed that the size of these corner voids in the horizontal orientation could be reduced by lengthening the distributor.

Significance

Powder injection molding offers a unique net shape forming capability suitable for low-cost and high-volume production of high-performance ceramics, metals, and carbides. In addition, this approach can significantly reduce the amount of hazardous waste stream generated in electroceramic, and potentially enhance the reliability of weapon components by elimination of surface machining induced damages.

Lead, used in electroceramic materials for power supply applications in the weapon systems, is a hazardous material. Currently, these lead-based ceramics come from sintered billets made by previous vendors. Standard machining processes are used to fabricate these ceramics into final geometries, which produces a large hazardous waste stream. Machining also increases labor costs by 18 percent and introduces surface and subsurface damages.

These minor damages can be exacerbated during a subsequent thermal incursion or a poling operation where a transformational strain or a field-induced strain associated with domain reorientation can induce additional crack propagation in these brittle ceramics. Sometimes, this can ultimately lead to component fracture and significantly impact production yield. For example, more than 20 percent of these components are lost due to cracking and chipping induced in the manufacturing processes.

These issues, common to many ceramic materials used in the piezoelectric industry, can be addressed by this newly developed injection molding technique – a near-net-shape forming process that will reduce labor costs, minimize waste stream (at least 90 percent), efficiently use raw material, enhance production yield, and increase the reliability of ceramic-based weapon components. Furthermore, with old raw materials becoming depleted, the development of new powder synthesis and ceramic forming capabilities is critical.

Most defects generated by injection molding, such as knit lines and cavities, can be eliminated by proper mold design and control of material flow. The location of the gate relative to the component cavity is equally important in producing high-quality parts. A properly placed gate can reduce turbulent flow, quickly filling the mold cavity, and help join the flow fronts into a monolithic part. To date, the optimization of the mold design and injection molding process has been essentially experimental in nature.

Based on our rheological study, we have developed 2D (for multiphase, nonNewtonian fluids) and 3D (Newtonian fluid) computational simulation capabilities for the flow modeling to determine the processing and tooling parameters. With these new capabilities, we can design and manufacture injection molds for other components more efficiently and without extensive iteration. Together with a statistically based design of experiment approach for optimizing molding parameters, a science-based engineering process has been developed at Sandia for injection molding of high-performance ceramic components.

Our promising results have led to subsequent funding from the Process Development Program for net-shape forming of varistor materials, and the knowledge gained from this effort will pave the road for future advanced application development.

Other Communications

R. Rao, L. Mondy, D. Noble, P. Notz, T. Baer, M. Hopkins, L. Halbleib, A. Grillet, and P. Yang, "Level Set Modeling of Injection Molding," presented at the 15th US National Congress on Theoretical and Applied Mechanics, Boulder, CO, June 2006.

Macro-Meso-Microsystems Integration in LTCC

67008
K. A. Peterson, K. D. Patel, C. K. Ho, C. A. Walker, B. R. Rohrer, S. B. Rohde, C. D. Nordquist, A. L. Casias, B. Wroblewski

Project Purpose

Low-temperature cofired ceramic (LTCC) technology is at the center of a new interest in meso-macro-micro (MMM) scale system integration. In addition to being exploited for new applications in silicon microelectromechanical systems (MEMS), LTCC has carved out a niche in the mesoscale that is necessary and unique from silicon. The versatility and manufacturability of LTCC at low cost is attracting unprecedented interest.

The purpose of this project is to exploit new capability by implementing new 'outside the box' techniques to make structures never previously realized that enhance the capability of microsystems.

Microsystem integration involves technologies as varied as integrated digital logic, radio frequency (RF), optics, microfluidics, a host of sensors and actuators, and chemical and physical analysis. LTCC technology can meet requirements for critical microsystem elements due to its excellent high-frequency properties, nearly unlimited stacking capability, chemical inertness, and form and fit versatility.

The purpose of this project is to demonstrate solutions to microsystem integration focusing on the advantages of LTCC. Its limitations include low thermal conductivity, special materials needs for embedded passives, and low strengths associated with the attachment of electrical leads. With new innovations, previous drawbacks to the technology can be overcome.

LTCC has proven to be an enabling medium for microsystem technologies because of its desirable electrical, physical, and chemical properties and coupled with its capability for rapid prototyping and scalable manufacturing of components. LTCC is viewed as an extension of hybrid microcircuits, and in that function it enables development, testing, and deployment of silicon microsystems. However, its versatility has allowed it to succeed as a microsystem medium in its own right, with applications in non-microelectronic mesoscale devices and in a range of sensor devices. Additional applications for cofired transparent windows, some as small as an optical fiber, have also been achieved.

FY 2006 Accomplishments

We developed processing techniques to generate new structures. These include the use of various sacrificial volume materials (SVM) to create moving parts, sensor cavities, microfluidic channels and suspended thick films, and the use of novel forming and fabrication techniques to obtain structures such as rolled tubes. Some of the other structures we successfully demonstrated include cofired strain gages and pressure sensors, microfluidic multichip modules, analytical and functional microfluidic parts such as a biological cell lyser, and smart channels with the capability to detect various chemical agents.

Accomplishments include silicon microfluidic 'chip-and-wire' systems and fluid grid array (FGA)/ microfluidic multichip modules using embedded channels in LTCC, and cofired electromechanical systems with moving parts. Both the microfluidic and mechanical system applications are enabled by sacrificial volume materials (SVM), which create and maintain cavities and separation gaps during the lamination and cofiring process. SVMs consisting of thermally fugitive or partially inert materials are easily incorporated.

Screeding is an incorporation technique that improves uniformity and eliminates processing steps. As developed in this work it is a self-aligned processing mask for placing sacrificial material in specific patterns. Recognizing the premium on devices that are cofired rather than assembled, we fabricated functional-as-released and functional-as-fired moving parts for the first time, including an impeller that has been exercised over thirty million cycles and a cofired pressure sensor that requires only pressure source and electrical connections. Cofired transparent windows, some as small as an optical fiber, were also developed and verified to be hermetic.

Significance

The work of this team has elevated the state of the art in LTCC in the areas of incorporating enclosed, unfilled volumes for use in microfluidics, integral components and sensors, and analytical hardware. Our work last year sparked considerable university and industrial interest, both domestic and international, at an April 2006 ceramics conference.

In particular, the novel use of SVM (patent pending) has created new capabilities for complex microsystem boards. We gave presented examples of these structures in several applications where LTCC has been essential to microsystems and mesoscale sensors. This work has been cited by international authors, and resulted in three invited publications, as well as an invitation to give an invited talk at the Poland Chapter of the International Microelectronics and Packaging Society in September, 2007.

This work is relevant to Sandia programs, including new flexible radar multichip module concepts that will use LTCC boards to establish high reliability systems at low cost. This radar work depends on embedded components.

Microsystems present a vast array of capabilities, including electrical, RF electrical, optical, fluidic, and electromechanical phenomena, often implemented in what are known as microelectromechanical systems (MEMS). Many of these systems involve silicon devices, many of which are fabricated by surface micromachining techniques on layers that are stacked within several microns of the surface. They enable biological and life science analyses, optical switching and beam modification, wireless communication, and many developing applications. Frequently, the advantage is lost if they are not integrated in a way that preserves miniaturization.

LTCC can meet many integration needs while bringing well-known advantages, including high-conductivity electrical traces, low dissipation factor dielectrics, inherent manufacturability, high-temperature operation, chemical inertness, and versatility in integrating multiple interrelated devices in a variety of critical proximities, orientations, and environments. This integration can be accomplished with superior performance at a reasonable cost.

Refereed Communications

K.A. Peterson, K.D. Patel, C.K. Ho, B.R. Rohrer, C.D. Nordquist, B.D. Wroblewski, and K.B. Pfeifer, "LTCC Microsystems and Microsystem Packaging and Integration Applications," Journal of Microelectronics and Electronic Packaging, vol. 3, pp. 109-120, 2006.

C.K. Ho, K.A. Peterson, L.K. McGrath, and T.S. Turner, "Development of LTCC Smart Channels for Integrated Chemical, Temperature, and Flow Sensing," in Proceedings of the 2nd International Conference on Ceramic Interconnect and Ceramic Microsystems Technologies (CICMT), April 2006, CD-ROM.

K.D. Patel, K.A. Peterson, K.W. Hukari, and T.S. Turner, "Low-Temperature Cofired Ceramic Microfluidic Microsystems for High-Temperature and High-Pressure Applications," Journal of Microelectronics and Electronic Packaging, vol. 3, pp. 152-158, 2006.

Design and Manufacture of Complex Precision Optics

79741

D. D. Gill, A. A. Claudet, V. C. Hodges, M. J. Vasile

Project Purpose

The miniaturization of security and defense systems is creating a need for high-precision micro- and mesoscale optical systems. This is especially true in microrobotics where navigational vision systems must occupy minimal space, return maximum resolution, and be inexpensive to manufacture. The ability to produce such optics is a new capability for Sandia using the Moore 350FG Freeform Generator and Wire EDM (electrical discharge machine).

We investigated the design, production, alignment, and metrology of complex refractive/diffractive hybrid optical systems including design methodologies that integrate the manufacturing capabilities of these precision processes. The complexity of the optics was beyond the capability of current processes and necessitated the development of new process enhancements and integration of multiple manufacturing processes. These capabilities have enabled Sandia designers to greatly increase the complexity of designs while reducing the overall size for both nuclear weapon (NW) and nonNW systems.

We developed a revolutionary micro-optical, multilayer, dragonfly-eye microlens array for microrobotics. These microlens arrays are designed to provide very good imagery over a >150 degree field-of-view. The array is extremely compact at 4 mm diameter and 2 mm thick. The system consists of two concentric domes and a coherent fiber bundle. One dome contains the diffractive/refractive lenslets and the other is a spacer/field stop. The fiber bundle transfers the curved image to a flat CCD (charge coupled device). Fabrication of the lenslets and registration, front-to-back, will be the greatest challenges.

We developed manufacturing and design technology for a multilayer array of precision hybrid diffractive/refractive micro-optics, and for precision alignment and mounting components produced by EDM.

FY 2006 Accomplishments

We developed the means to fabricate the highly complex dragonfly eye optic. This included a research effort to modify spherical diamond mills into aspherical mills using a focused ion beam for nanometer precision machining. The technique required the tool to be prepared with a sacrificial metal coating to prevent rounding from the beam waste, to be precisely mounted in the focused ion beam machine, and to be imaged using the secondary electron emission.

We used a computer-based vision processing method to calibrate the image size to real-world units, and completed a mask definition that specified the material to be removed from the sphere to achieve an asphere. We loaded the mask definition into the controller of the focused ion beam machine and used the focused ion beam to erode the unwanted material from the tool edge. We removed the sacrificial material and measured the tool.

We then used this tool on the Moore 350FG precision machining center to plunge mill-precision, convex aspherical optics around the periphery of a convex polymer dome. The plunge milling of these optics required the development of high-speed displacement measuring capability to reduce the radial rotational error from the tool motion. We developed a kinematic mounting system for mounting the high-speed spindle on an ultrahigh-repeatability rotary table. We developed a method for aligning the plunge mill with the turned optic and created an optic with many lenslets on the surface.

In this process, we developed a novel method for machining the optic from a larger polymer substrate, which achieves the significant repeatability requirements for the optic and allows precision positioning of the optic in the machine. This technique has since been applied to other very small optics in follow-on projects.

As part of this research, we developed a method of measuring parts in the diamond turning machine using a laser form interferometer. This capability has allowed the measurement of mesoscale and macroscale optical arrays with high accuracy over the range of the machine tool.

Significance

This research has directly influenced the fabrication and design of high-precision, mesoscaled optics at Sandia and has provided new methods of positioning, locating, and machining these tiny optics with high precision. The modification of concave diamond mills has allowed the creation of off-axis, precision, aspherical microlenses arrayed on 3D surfaces, which has never been available in the past.

The fixturing and location methods developed in this work have allowed the fabrication of several optics that would not have been possible in the past at Sandia. These methods are being applied to optics that meet the needs of a multitude of customers.

The project has resulted in significantly more collaboration between several organizations at Sandia, providing a much better understanding of what the organizations do and therefore a better use of their expertise.

This project also provided an opportunity for the manufacturing group to probe the limits of current processes and techniques and to communicate the boundaries of the manufacturing space to the optical designers. Armed with this knowledge, the optical designers are now able to design optical systems that can realistically be fabricated, and thus the customer is presented with a good estimation of the final optical performance of the system before tools cut parts.

Other Communications

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D.D. Gill, W.C. Sweatt, A.A. Claudet, D.P. Adams, V.C. Hodges, and M.J. Vasile, "Aspherizing the Sphere: Using Focused Ion Beam Machining to Create Aspherical Diamond Mills for Micro-Optics," in *Proceedings of the Annual Meeting of the American Society for Precision Engineering*, October 2006.

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Development of a Manufacturing Capability for Production of Ceramic Laser Materials

79742
T. J. Garino, E. D. Spoerke, L. S. Weichman, R. L. Schmitt, J. Cesarano III, S. J. Lockwood, P. Yang

Project Purpose

The purpose of this project is to develop the capability to fabricate transparent ceramic laser host materials. We focused on two materials: yttrium aluminum garnet (YAG) and gadolinium scandium gallium garnet (GSGG). We chose YAG because it is the most important solid-state laser host material and because there has been extensive previous work in this area that we can build upon. We chose GSGG because, unlike YAG, it is a radiation-hard laser host, which makes it suitable for weapons applications.

As opposed to single crystals that are grown from a melt at very high temperature, transparent ceramic materials are fabricated by solid-state sintering of fine powders at significantly lower temperature. The challenge is to achieve a much lower level of porosity and contamination than is normally produced by sintering of ceramic powders so that the material will have the desired optical properties.

Polycrystalline ceramic laser host materials have important advantages over traditional single crystal materials. Single crystals, especially of GSGG which is available only by special order from only one supplier, can have a variety of defects such as dislocations, spiral growth, and cracks; contamination such as iridium inclusions from the crucible; and inhomogeneities in dopant distribution.

These types of defects and contamination are not present in polycrystalline ceramics, in which dopant levels and distribution can be more precisely controlled. Polycrystalline ceramics can also be direct-formed into complex shapes or with controlled gradients in dopant concentrations using techniques such as Sandia-developed robocasting, attributes that are difficult or impossible to achieve with single crystals.

To produce transparent polycrystalline samples of these materials for laser host applications, we must first synthesize high-purity, active powders of both materials that have the proper composition and contain the desired levels of dopants: Nd for YAG and Nd and Cr for GSGG. The powders must then be processed into usable shapes with uniform microstructure without introducing contamination. Finally, these samples must be sintered in a controlled manner so that the final porosity will be in the parts-per-million range, with a grain size in the micrometer range, without introducing any contamination.

FY 2006 Accomplishments

Fabrication of transparent YAG achievements:

Powder Synthesis

- Transferred batch production of the YAG powder to the Ceramics and Glass Processing Department
- Greatly improved the control of the aluminum-to-yttrium ratio in our as-synthesized powders so that we can effectively eliminate light scattering second-phase particles of YAlO3 in our sintered materials
- Improved the post-synthesis processing of the YAG powder so that it is more easily dispersed.

Powder Processing

- We were able to achieve the desired degree of dispersion of the YAG powder using only a sapphire-tipped ultrasonic horn, which decreases the contamination of the powder compared to when it is milled using grinding media.
- We achieved another process improvement that decreased contamination by using vacuum casting instead of slip casting on plaster molds to form YAG samples from aqueous slurries.
- We increased the starting density of our unfired samples significantly by cold isostatic pressing, which leads to improved sintering behavior.

Direct Fabrication

- We performed the first successful direct fabrication of complex shapes of YAG by robocasting an aqueous YAG slurry using our chemically-prepared powder.

Sintering

- We greatly decreased the contamination of the YAG samples during high-temperature sintering by using coarse YAG powder to surround the pellets during sintering and by using only a clean high-temperature furnace.
- Based on the results of our sintering experiments, we modified our sintering schedule to eliminate the large pores that were present in earlier samples and to better control the final grain size of the sintered material.

Fabrication of transparent GSGG accomplishments:

Powder Synthesis

- We demonstrated the first chemical synthesis of Nd and Cr doped GSGG precursor powders with the desired composition of the five metals using a process similar to that used to synthesize the YAG powder in which ammonium carbonate is used to precipitate the precursor powder from a metals nitrate solution.
- We found that adjusting the pH down to a specific range during the synthesis reaction was crucial to forming a precursor that contained the stoichiometric amounts of all the metals and that would form the garnet phase after calcining to 1000 ºC.
- We characterized this powder and determined that it has desirable uniform, nanosized particles.

Powder Processing

- We processed this powder to form pellets without introducing contamination by using a sapphire-tipped ultrasonic horn to mill the powder to the desired size and then vacuum-casting the aqueous slurry to form uniform green bodies.

Sintering

- We characterized the shrinkage behavior of our GSGG powder using optical dilatometry and then demonstrated that a relative density of 95 percent could be reached by sintering to 1500 ºC for two hours.
- We produced the first reported translucent ceramic GSGG by vacuum sintering to 1650 ºC.

Significance

Fabrication of Transparent YAG:

- The ability to produce large batches of stoichiometric, dispersible YAG powder. Having a reproducible process for making powder with the desired characteristics is critical for both making progress towards fabricating transparent material as well as having success in any future production applications.
- The ability to form the YAG powder into shapes with fine, uniform microstructure without introducing contamination. A technique to form the powder into a useful shape with a uniform packing to particles and minimal defects or contamination is also a necessary step toward producing a transparent ceramic laser host.
- The ability to direct-fabricate complex shaped parts with the YAG powder. This is, to our knowledge, the first time anyone has demonstrated this ability with YAG powder, and it is a key step toward fabricating novel laser structures for micro-optics or with graded dopant profiles.
- The ability to sinter the YAG powder without introducing contamination of forming large pores. Though previous samples have been "transparent," they contained large pores of second-phase inclusions that once present are impossible to remove from the sintered material. Thus, we are on the verge of being able to, with minor sintering schedule modifications, eliminate the small amount of micrometer-sized pores still present to achieve transparency.

Fabrication of Transparent GSGG:

- Demonstrated the synthesis of Nd and Cr doped GSGG powder composed of uniform nanoparticles. The synthesis of codoped GSGG has not previously been reported. Without a starting powder with the desired composition, phase, and particle-size distribution, fabrication of a transparent ceramic GSGG laser host would not be possible. This accomplishment was aided by the knowledge and experience gained from our work on YAG.

- The ability to form the GSGG powder into shapes with fine, uniform microstructure without introducing contamination. A technique to form the powder into a useful shape with a uniform packing to particles and minimal defects or contamination is also a necessary step toward producing a transparent ceramic laser host. This accomplishment was aided by the knowledge and experience gained from our work on YAG.
- The first sintered GSGG powder sample was translucent. This accomplishment, also one that has not been previously reported, is significant because it gives us confidence that only minor modifications of the sintering time/temperature profile will yield transparent material. Once again, what we learned from our work with YAG benefited our work with GSGG, allowing us to more quickly progress.

Large-Scale Manufacturing of Integrated Nanostructures for Sensing

93426

S. L. Shinde, T. P. Swiler, D. P. Adams, J. W. Hsu, G. D. Bachand, E. D. Spoerke, B. C. Bunker

Project Purpose

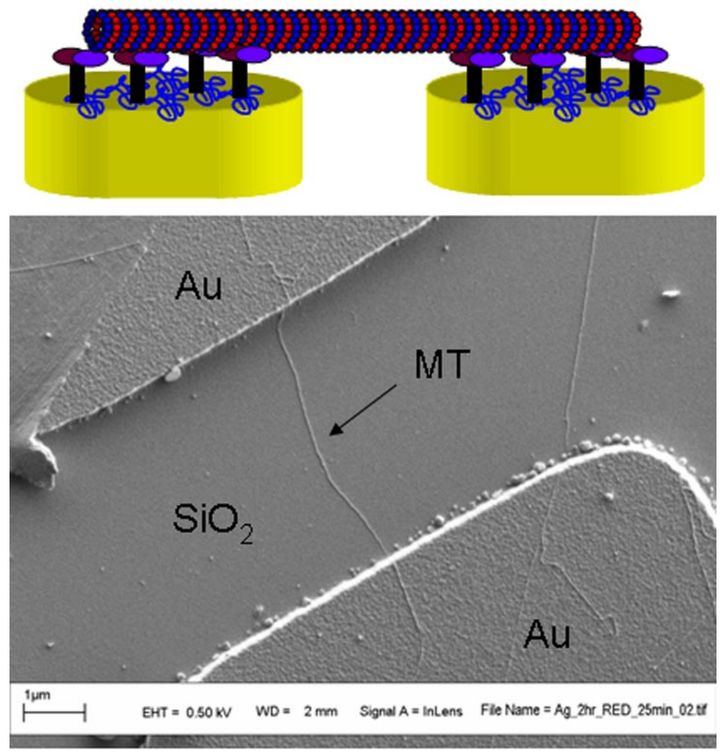
Nanostructures have great potential for providing integrated function for very high-density sensing, interconnects, packaging, and other microelectronic applications. The materials community has produced numerous nanoparticles, nanowires, nanotubes, and nanobelts but lacks methods to incorporate these nanomaterials into useful, functional devices. Methods such as flow-induced patterning and individual nanostructure nanomanipulation have been explored, but these approaches are difficult to control and are not scalable, problems that significantly limit the widespread application of those approaches.

The purpose of this project is to develop a completely different approach for large-scale manufacturing and assembly, combining top-down lithography with an innovative bottom-up approach. Specifically, our work integrates lithographic micropatterns with cooperative biomolecules, such as motor proteins and microtubules, as nanocargo transport and positioning apparatus.

We use chemical strategies to drive the self-assembly of microtubule scaffolding networks on lithographic micropatterns. By properly activating or deactivating the biomolecules, cargo (e.g., nanoparticles and nanowires) can be rapidly positioned, assembled, or templated in potentially large systems. As a new manufacturing process to assemble functional nanodevices, this revolutionary approach has real potential to overcome major obstacles in nanotechnology.

FY 2006 Accomplishments

We developed methods for the selective functionalization of patterned surfaces and use of these techniques to capture microtubules (MTs) on these patterns. We used selective chemistries to discriminately functionalize micropattened silica and gold. We then demonstrated that this chemical treatment effectively allowed casein-mediated kinesin motor protein attachment only on the gold structures.



A captured microtubule is visible in this scanning electron micrograph forming a nanoscale bridge (arrow) between two gold pads. This arrangement is schematically depicted in the cartoon above where a microtubule bridge is captured on two gold pads (yellow) by kinesin motor proteins selectively bound to the gold pads.

These attached motor proteins, then, captured polymerized MTs out of suspension through the specific, cooperative biological interactions inherent to these cooperative biological agents. We then used this strategy to create MT bridges between lithographically-patterned gold features.

We established chemical approaches to metallize MT arrays, creating functional interconnects. We chemically modified the MT bridges spanning gold microstructures to create functional metallic silver bridges between the gold pads. We bound aqueous silver ions (AgNO3) to the MT protein structure and chemically reduced it, nucleating a coating of polycrystalline silver along the MT bridges. In a fluorescence micrograph of a silver-coated MT bridging two kinesin-coated gold lines, we observed red fluorescence emitted from the silver nanoparticles. Basic measurements of electrical connectively showed a resistance across this Ag/MT bridge of 500-600 Ω.

We established processes to attach nanocargo to kinesin motors and demonstrated kinesin-based transport of this cargo. We also successfully developed a strategy for attaching various micro- and nanocargo to N-terminal kinesin motor proteins. Adapted from the methods used to capture kinesins on the gold described above, our approach uses casein proteins to mediate the assembly of kinesin motor proteins onto cargo surfaces such as silica microspheres, functionalized polymer microspheres, and ZnO nanoparticles.

To the best of our knowledge, this work represents the first time ZnO has been successfully transported using kinesin motor proteins. Future work will combine these efforts with the organized MT networks described above to create an active cargo-positioning technique.

We designed a lithographic testing platform that allowed us to probe the electrical properties of our interconnect structures. This platform comprises gold lines raised several microns above the silicon surface, forming a linear "mesa." As MTs are captured to form bridges across the gaps in the test structure, we can use conductivity probes to measure their properties through connected test pads.

We carefully designed the geometry and chemical composition of these structures so as to optimize MT bridging with the least probability of interdevice "shorting" or other undesired surface-based interactions. This complex lithographic assembly required the development of a new series of processes for fabricating isolated, self-aligning mesa structures. Using a single photoresist approach, we used a combination of etching techniques to carve the platform structures out of a multilayer film.

Significance

Our results represent a number of significant steps toward meeting a key demand of nanotechnology – the integration of nanomaterials into multifunctional systems. Continued development of these methods has tremendous potential implications both for Sandia and the scientific community as a scalable system for integrated sensing and microelectronic miniaturization, intelligent systems for national security, and energy management.

More immediately, this project has provided us with a new set of technical skills and capabilities that will prove useful for a number of programs, such as a Basic Energy Sciences-funded program on active assembly (a biointegration project). Success in this project is also expected to be of great interest to users and staff in the Center for Integrated Nanotechnologies as well as customers in electronic packaging, advanced manufacturing, and sensor development.

Other Communications

E.D. Spoerke, G.D. Bachand, A.M. Trent, B.C. Bunker, J. Liu, C.E. Warrender, A.M. Bouchard, and G. Osbourn, "Biologically-Directed Nanoscale Materials Assembly," presented at MRS Spring Meeting, San Francisco, CA, April 2006.

Titanium Cholla-Optimized, Lightweight, High-Strength Structures for Aerospace Applications

93491

D. D. Gill, E. G. Winrow, A. A. Claudet, J. Robbins, C. J. Atwood

Project Purpose

Aerospace and weapons applications demand the lightest possible high-strength, high-stiffness structures to maximize payload efficiency while structural rigidity and component alignment are precisely maintained. If advanced weapons and aerospace applications are to be realized, cutting-edge structure optimization methodologies and manufacturing technologies are needed. These structures, which include optimized trusses, rib-on-plate housings, and three-dimensional (3D) aerospace structures, were not manufacturable prior to the development of 3D additive manufacturing processes like Laser Engineered Net Shaping (LENS).

This project is a joint effort for the design and manufacture of these optimized structures. We are working with Drs. Dewhurst and Taggart of the University of Rhode Island to use their newly established optimization algorithms and adaptive finite element-based topological optimization and to develop the X-FEM (extended finite element methods) as an extension of advanced topological optimization (ATO) schemes.

We are using mechanical modeling techniques in conjunction with the optimization algorithms to develop optimized solutions for rib-on-plate and 3D structures under multiple load conditions. We will develop methods to create the optimized structures by extending LENS to full 3D capability, including the development of 3D additive process planning. We expect to complete samples of optimized 2½-dimensional truss and rib-on-plate aerospace structures, and 3D structures including multiply-loaded, optimized structures inspired by cholla cacti.

This research will create the methods for optimizing lightweight, high-strength, 3D structures for aerospace and weapons applications. Though the basis for both the optimization and the layer additive manufacturing methods exist, the proposed move to 3D, multiply-loaded structures, and the manufacturing process and planning required to make these structures, is a significant challenge. With this challenge comes the opportunity for significant rewards through important, publishable work with the potential to reduce flight weights of structural components by 30-50 percent while maintaining the same strength.

FY 2006 Accomplishments

The project team made significant progress in the development of schemes for optimizing structures and the manufacturing capability required to realize these structures. We focused our efforts thus far on selecting case study parts, improving the efficiency of the University of Rhode Island's 2D ATO, developing the ability to use unstructured meshes in Sandia's X-FEM code, and completing the design, specification, and procurement of two additional axes to make the LENS process capable of building fully 3D structures.

We obtained parts for use as case studies from a potential user of the technology. These technology demonstration parts will allow us to solve real problems while demonstrating the valuable optimization capability. Our enhancement of ATO/density-FEM included an improvement in which the density of every element is changed with every iteration. This improvement significantly increased the speed of convergence but did not diminish solution stability because of an Euler-like method for controlling the density change.

Enhancements to the X-FEM capability have included the addition of unstructured meshing. The unstructured mesh allows portions of components to remain outside the mesh so that they are recognized as not being eligible for optimization (i.e., bolt holes, fixed geometry, and so on).

We also integrated ATO into the X-FEM code such that X-FEM can be used to analyze strain fields in components that are being optimized. Optimized 3D parts will require a fully 3D capable additive process to manufacture. LENS currently has limited 3D capability, but the team designed and ordered two additional machine axes giving the process full 3D motion. Additionally, we developed the initial plan of attack for the complicated task of 3D process planning using medial axis transformations as a core technology.

Significance

The results of this year's efforts will significantly impact structural applications both within and outside the nuclear weapons complex. The significance of the results is in three areas, matching the three parallel activities of this research. The first is the adaptation of ATO to multiple loadings and refinement of the code to increase speed. The impact of these advancements will be in permitting the optimization of 2D structures that may experience multiple, unique loading cases.

Because these loading cases cannot be solved for merely a superposition of the multiple cases, this newly developed method will allow the optimization of aerospace structures that have been, thus far, over-designed to accommodate these multiple loads. In addition, the code refinement has allowed the speed of the ATO solution to increase greatly and still maintain stability in demanding testing.

The second significant development is the introduction of unstructured mesh capabilities to the X-FEM. This capability allows regions of a part to remain unavailable to the optimization routing, thus preserving bolt holes, and so on.

The third development is the addition of a 4th and 5th axis to the LENS machine, allowing full 3D capability. This addition will have a significant impact because it will allow the manufacturing of complex 3D parts that haven't been available before. It will also speed the process for building parts since a support structure will no longer be necessary to support overhanging structures.

In Situ Optical Diagnostics of Neutron Generator Target Films

93492

M. B. Sinclair, M. E. Schendel, J. H. Flemming, W. R. Wampler, C. S. Snow, D. P. Adams

Project Purpose

Neutron generators employ two types of metal-hydride films, known as occluder films, that act as storage reservoirs for the tritium and deuterium required for the neutron production process. The ion source occluder is a thin ScD_2 film on a cermet substrate, and the target occluder is an ErT_2 film on a molybdenum or kovar substrate. Proper operation of the neutron tube requires that the stoichiometry of the occluder films be tightly controlled.

In this research project, we will develop in situ optical probes for determining the hydrogen isotope to metal ratio of occluder films. The optical diagnostics will exploit two important characteristics of these metal-hydride films. First, the inherent optical properties of these films depend sensitively on the hydrogen/metal concentration ratio, ranging from metallic (Drude) behavior at low hydrogen concentrations to the transparent insulator properties of the trihydride.

The desired dihydride stoichiometry behaves as a metal but possesses a pronounced optical transmission window in the red to near-infrared region. These dramatic changes in optical properties can be monitored using a number of techniques, including absorption, reflectance, and ellipsometry, and can be used to provide a measure of the current status of the occluder films.

Second, the state of film stress depends upon the hydrogen isotope to metal ratio as well as a number of other factors. In this case, optical stress measurements using substrate curvature techniques can provide the desired information.

We will identify the optimal architecture for performing each of these measurements in situ, with an ultimate goal of measurement of occluder film stoichiometry within the loader. In addition, we will investigate several fundamental aspects of the behavior of these materials including the relationship between residual stress and optical properties, as well as the existence of new stress-stabilized phases.

FY 2006 Accomplishments

The portion of this project directed toward the measurement of optical properties of ErH_x films is on-track, and our milestones have been achieved. During the first phase of this project we measured the optical properties of high-quality ErH_x films to establish the baseline optical properties of these materials. Specifically, we performed variable angle spectroscopic ellipsometry measurements on 11 different ErH_x films with loading levels that varied from $x = 0$ to $x = 3$. Thus, this set of films spans the range from the pure erbium metal, through the alpha phase (hydride), the beta phase (dihydride), and all the way to the gamma phase (trihydride).

In addition to the optical measurements, we performed x-ray diffraction and ion beam analysis on these films to determine their crystallographic structure and loading ratio. Our results showed clearly discernable differences in the optical properties of films with different levels of hydrogen loading, indicating that it should be possible to develop an optical probe that is capable of performing stoichiometry measurements.

In addition, we observed “optical windows” in the red to near-infrared spectral ranges in which optical beams can penetrate relatively deeply into the target films. The existence of these windows will allow us to develop probes that can measure the bulk properties of the target films.

Significance

Sandia is responsible for the design, production, and health of neutron tubes for the entire stockpile. Significant effort is needed to understand the materials properties of all components found in the tube and generator. If successful, this project will develop a novel optical probe that will enhance our ability to steward the nuclear weapon stockpile by improving our ability to ensure the reliability of neutron tubes. The nondestructive stoichiometry measurements will provide truly nondestructive acceptance testing of all target lots; a novel analytical tool for a wide variety of applications such as monitoring target films during hydriding, conditioning, and throughout deployment; and an independent determination of phase structure.

All of these will provide insight not previously available to the design and production community. Success could produce a concomitant reduction in production cost and increase the reliability of the product through a better understanding of design margins to prevent catastrophic failures of the tube.

Other Communications

M.E. Schendel, C.S. Snow, C.R. Tewell, D.P. Adams, S.H. King, M.B. Sinclair, and M.O. Eatough, "Rapid Nondestructive Evaluation of Neutron Tube Targets," presented (poster) at the NNSA Future Technologies Conference II: Technology as a Catalyst for Enterprise Transformation: Trends & Strategies, Washington, DC, October 2006.

Low-Cost, Mesoscale Parts Fabricated from Nanocrystalline Metals

93493
D. D. Gill, P. Yang, T. J. Vogler, A. C. Hall

Project Purpose

Nanocrystalline metals have very high strength and can be made to have good ductility as well. The capabilities of mesoscale surety device components could be expanded if the parts were produced from these nanocrystalline metals. However, the means of producing nanocrystalline metals create powdered material that must be combined in some manner that prevents grain growth.

The purpose of the project is to discover techniques by which nanocrystalline metals in powder form can be compacted into bulk form and used to fabricate mesoscale surety components. The project team is investigating several means of producing nanocrystalline materials, including ball milling and severe plastic deformation. These techniques produce nanocrystalline powder, but this powder is difficult to compact into bulk material so that it can be used in demanding applications.

We are investigating cold spray and shockwave compaction as means of forming bulk materials without inducing grain growth in the material and subsequent loss of material properties. We are testing the bulk materials to determine properties and machining characteristics, and conducting performance tests on the parts.

In addition, it is possible to create bulk nanocrystalline material in the form of “foils” or chips produced by the machining process. By controlling the process parameters, it is possible to create nanocrystalline foils that are relatively flat with good surface finish.

We are working to understand the mechanics of creating foils, the means by which anisotropy, residual stress, and surface finish can be controlled, and methods for machining parts from these foils using electro-discharge machining. The foils are not large, but they are large enough to produce mesoscale devices, which will be tested to determine performance capabilities and characteristics.

FY 2006 Accomplishments

We established a new capability to fabricate nanocrystalline metal powder (6016-T6 aluminum alloy) by the modulation-assisted machining (MAM) process. In addition, we made our first attempt at low-temperature consolidation of nanocrystalline aluminum alloy (cryogenically milled 5083 aluminum) by the cold spray process, completed our mold design, and performed preliminary simulation for the shockwave consolidation process. Furthermore, we fabricated mesoscale demonstration parts (with a minimum feature size of 40 μm) and tensile test specimens directly from a severe plastic deformed Inconel 718 foil.

This process, using a deep cut approach, can produce large-sized foils that are suitable for the fabrication of mesoscale components. Transmission electron microscopy and x-ray analysis indicated that these foils possess nanocrystalline structure with significant amounts of built-in microstrain (strain broadening ranges from 0.3 percent to 0.7 percent, depending on the planes in calculation). However, there is no evidence of texture formation, considering these foils have been plastically deformed. Preliminary results showed an increase in yield strength but a decrease in ductility, which is commonly observed for nanocrystalline metals.

We will explore an additional thermal treatment to increase the ductility and enhance the mechanical performance. This new approach, if successful, will significantly reduce the manufacturing costs and shorten the production cycle.

We will continue to refine our newly established MAM system to control powder morphology and size, perform several low-temperature consolidation experiments for both cold spray and shockwave compaction processes, and prepare for microstructural and mechanical property evaluations next fiscal year.

Significance

The proven ability to create mesoscale parts directly from foils produced by severe plastic deformation offers the opportunity to create high-strength parts for demanding applications. There are still issues to be addressed, but the ability to create surety components with the unique material properties of nanocrystalline materials would open a whole new set of opportunities for designers of these components. Additionally, the relatively low cost of creating and using foils would position them as an economical alternative to other specialized, high-strength materials.

The ability to form bulk, near-net shape nano-crystalline material by cold spray and shockwave compaction will give designers the opportunity to use these materials in macroscale applications as well. Though there is still a lot to learn about the compaction of the materials, the first year's results indicate that the methods are promising for the creation of bulk nanocrystalline metals.

New, Low-Cost Material Development Technique for Advancing Rapid Prototyping Manufacturing Technology

93494

D. D. Gill, P. M. Booker, E. A. Holm

Project Purpose

The purpose of this project is to develop a new material synthesis methodology using additive manufacturing techniques such as Laser Engineered Net Shaping (LENS) to create new production-quality materials for rapid manufacturing and response to evolving threats. Conventional alloy development requires years, consumes millions of dollars, and requires large heats of material that are scrapped at the end of the development cycle.

We are adapting a methodology used to evaluate new titanium alloys for an Air Force Materials Laboratory (AFML) project and using it to evaluate iron-based alloys. This is enabling us to create new classes of composite materials important to meeting future weapons needs for rapid response and hard target defeat.

We are also using a laser-based additive manufacturing technique to investigate entire alloy composition spectra with small numbers of samples in a condensed time frame wasting little material. By mixing different constituents under precise computer control, we can vary composition on a millimeter basis over a complete spectrum of alloy compositions to create 5 cm-long graded composition samples. These samples are ideally suited for sophisticated materials analysis, including scanning electron microscopy for phase identity, volume fractions, and phase boundaries, resulting in a complete phase diagram study.

Once a suitable composition is determined, the same additive manufacturing technique will make small-lot production of near-net-shape parts. Example materials include steels that do not require heat treatment but have increased blast resistance, stiffness features, embedded hollow particles in the matrix creating low-density, reticulated structural materials from conventional high-density alloys, or hard dispersoids to control grain size and increase wear resistance.

FY 2006 Accomplishments

This year's accomplishments include:

- Selection of a candidate binary material system
- Acquisition of metal powders
- Recovery of a multimaterial depositing capability for LENS
- Initial analysis of the material
- Deposition of material samples from the candidate materials

The material system we selected is an iron-manganese system that offers good strength and ductility characteristics with very fast work-hardening under surface abrasion. The material family is referred to as Hatfield steels and has application in penetrators. Because of the fast work-hardening, the material is difficult to machine, making it an excellent choice for near-net-shaping by LENS. We chose a binary system to simplify modeling and analysis while the team is developing the processes to model, create, and study the material samples.

The recovery of a multimaterial depositing capability for LENS includes an ongoing effort to update software to run on current hardware platforms, the reactivation of a powder feeder, and wiring of the feeder's controls. We deposited material samples with 304L stainless steel, iron, and manganese powders, followed by samples of 304L alloyed with varying percentages of iron. Metallography and melt-pool intensity analysis are ongoing for these samples to determine the effects of varying melt-pool properties on the closed loop melt-pool control system. The emissivity of the two materials is significantly different, so we are studying the effects of this difference on the melt-pool controller.

Significance

Our accomplishments thus far have been incremental and therefore do not have a large impact yet. However, these accomplishments will position the team to be able to make significant impact in reducing cost and accelerating the development time to create innovative alloys with the LENS metal-forming process.

Because the LENS can deposit fully dense, near-net shape parts in addition to coupons for materials evaluation and characterization, the new materials will then be immediately available for creating engineered parts with enhanced properties like high strength or high toughness. This will not only allow a whole new set of materials to be created, but will accelerate their implementation into demanding applications of all types for quick response to evolving threats.

Other Communications

J.E. Smugeresky, D.D. Gill, and C.J. Atwood, "Laser Engineered Net Shaping (LENS) as a Unique Process for the Repair and Modification of High-Rigor Parts," presented at NNSA Future Technologies Conference, Washington, DC, October 2006.

J.E. Smugeresky, B. Zheng, Y. Zhou, and E.J. Lavernia, "Process-Structure-Property Relationships in LENS Processed PH13-8Mo Steel," presented at 2006 TMS Annual Meeting, San Antonio, TX, March 2006.

B. Zheng, J.E. Smugeresky, Y. Zhou, and E.J. Lavernia, "Microstructure and Properties of PH13-8Mo Steel Fabricated by LENS," in *Proceedings of the 2006 International Conference on Powder Metallurgy and Particulate Materials*, pp. 10-81, June 2006.

Advanced Manufacturing of a Novel Functional Material

93495

C. B. DiAntonio, N. S. Bell, G. A. Samara, J. A. Voigt, M. A. Rodriguez

Project Purpose

Evolution in manufacturing science and technology does not necessarily need to manifest itself in modifications to existing processes. A quantum leap can occur by development and redesign of the product through a novel material and manufacturing procedure.

The prevailing target of this work is to develop an advanced, low-cost manufacturing process that results in a lead-free ferroelectric ceramic material with comparable properties to the presently used lead-based compositions, especially those that are widely used in the weapons program. Although there has been a concerted effort in the materials community to develop lead-free ferroelectric ceramics and a technique to synthesize them, no effective alternative to lead PZT has been found.

For a polycrystalline material, enhancements can be made to the anisotropic piezoelectric polar properties by fabrication of polycrystals that favor a preferred orientation. These oriented ceramics display improved and highly anisotropic properties when compared with randomly oriented ceramics. Many physical properties of crystalline materials are strongly dependent on the crystal orientation of the materials, and the potential benefits from this textural modification are not fully realized.

This work deals with the fabrication of a perovskite-type, nonlead-based polycrystalline ceramic through a template-induced texturing process using morphologically controlled platelets. The focus vehicle lies with the layer-structured perovskites, particularly the bismuth layered and alkali niobate-based solid solution ferroelectrics. We are using templated grain growth, or "reactive templated grain growth," to produce preferably oriented polycrystals.

We accomplish template synthesis by employing a fused-salt synthesis technique where molten salts are used as reaction aids to prepare complex oxides from their constituent oxides. The intent is that the use of templates and an appropriate forming process will result in fabrication of a functional material with enhanced dielectric, piezoelectric, and electromechanical coefficients.

We are investigating an innovative and low-cost material and fabrication process for the morphologically controlled templates, and an examination is under way on the correlations among texture development, the solid-state reactions, and microstructure-property relationships. The outlook for manufacturing advanced functional nonlead-based ferroelectric materials will be advanced with successful completion of this project.

A direct connection exists in this project to all of the strategic goals of the DOE, including:

- enhanced piezoelectric performance for improved sensing and actuation systems for national security
- more efficient fuel injectors to extend depleting fossil fuel reserves
- extended component stability, reliability, and surety due to texturing enhancements
- elimination of the environmental problem of the present technology's dependence on a 60 percent lead-containing family of ceramics

FY 2006 Accomplishments

As the project was initiated, the extent of literature information on the lead-free ferroelectric systems proliferated and allowed for an in-depth and up-to-date evaluation of potential systems to investigate. The main milestone was focused around selecting a candidate lead-free ferroelectric material system for study that, based on previous work, had the most potential as a lead-free ferroelectric alternative. At the same time a system that lent itself to textural modifications via microstructural engineering needed to be chosen for potential property enhancements.

The primary lead-free ferroelectric candidate system, $Bi_4Ti_3O_{12} – Na_{0.5}Bi_{0.5}TiO_3 – BaTiO_3$, was selected most-promising composition ternary from an exhaustive literature search. The system contains two perovskite end members – $Na_{0.5}Bi_{0.5}TiO_3$ and $BaTiO_3$ – and the third member, $Bi_4Ti_3O_{12}$, possesses the Aurivillius crystal structure.

This particular system was chosen based primarily on 1) detailed information on the crystallographic nature of compositions contained within the ternary, 2) the ability to couple seed templates of plate-like morphology $Bi_4Ti_3O_{12}$ to a texture-inducing forming process and ultimately produce a texture-engineered microstructure, and 3) the processing/synthesis space for this composition and derivative compositions has been previously mapped at a remedial level, although not fully resolved.

The study of a ternary composition also allows an increased sense of flexibility to not only examine tailoring a microstructure but to also explore compositional variations for tuning macroscopic properties. The team also selected a secondary system, strontium barium niobate (SBN), based on its literature-reported characteristics and potential for acicular seed growth in the unique ferroelectric tungsten bronze crystal structure. Although the seed morphology is considered "needle-like," the potential to induce a textured microstructure still exists.

We focused mainly on conventional ceramic powder synthesis (traditional mixed oxide route) and characterization of the individual members in the primary system of the ternary, $Bi_4Ti_3O_{12}$, $Na_{0.5}Bi_{0.5}TiO_3$, and $BaTiO_3$. We completed a first-order investigation for the powder processing and forming of the individual members, including processing, calcining, forming, and sintering parameters.

Thermal-gravimetric/calorimetric analysis provided detailed information on the decomposition of the processing organics, oxides, and carbonates and the kinetics of the reactions involved in phase development, including calcining and sintering profiles.

We performed x-ray diffraction and in situ x-ray diffraction analysis to verify composition and investigate the phase formation. The x-ray diffraction results confirmed that processing produced phase-pure individual members. Based on these results, two ternary phases were also fabricated: $0.33Bi_4Ti_3O_{12} – 0.33Na_{0.5}Bi_{0.5}TiO_3 – 0.33BaTiO_3$ and $0.0855Bi_4Ti_3O_{12} – 0.4815Na_{0.5}Bi_{0.5}TiO_3 – 0.433BaTiO_3$. In both cases, we verified two separate forming routes: a ternary end member synthesis and a raw material (oxides and carbonates) synthesis process. For both of these compositions, x-ray diffraction confirmed the desired mixed phase perovskite and Aurivillius structures.

In addition to successful mixed oxide powder synthesis of the desired compositions, we synthesized seed crystals of $Bi_4Ti_3O_{12}$ following a molten-salt synthesis technique.

Significance

Texture enhancement of a lead-free ferroelectric ceramic material may offer, for the first time, a bulk ceramic that is a viable alternative to the lead-based systems. Template seed crystals, used in the solid-state conversion, can significantly reduce the compositional inhomogeneity that tends to be inherent in other alternative methods, such as flux growth of bulk single crystals, and at the same time provide a lower cost, more manufacturable process.

The research and development in this project operates in a new region of synthesis space where the unique combination of morphologically controlled template particles and a forming process induce texturing in an otherwise isotropic material. The melding of these two technologies into one advanced manufacturing process is innovative. By harnessing the techniques of texture development and process optimization in developing an ideal and predesigned microstructure for enhanced material performance for a ferroelectric material, we have initiated the establishment of a robust, science-based manufacturing technology for advanced functional materials.

The present applications and emerging needs rely heavily on maintaining the momentum to develop low-cost advanced manufacturing processes. From a Laboratories' perspective, it is imperative that the ideals of lower costs and advanced manufacturing be intermingled in a novel process that produces a functional material that meets future needs. The proposed texture engineered lead-free ceramic material accomplishes both of those goals by not only enhancing materials properties but also eliminating the environmental and toxicity issues surrounding leaded compositions.

The establishment of this science-based manufacturing process would enhance our ability to provide improved capabilities for pulsed-power generation, advanced sensing technology for homeland security endeavors, superior sonar for the Navy, novel "smart" materials, and higher-resolution ultrasounds for medical diagnostics, to name only a few. The landscape for manufacturing novel advanced functional materials and enhancing existing components in the stockpile will benefit immensely.

Our FY06 accomplishments provided a strong foundation for fully realizing the possibility of microstructural engineering of advanced ceramics and having those advanced ceramics tied to a manufacturing process. The concept of textured non-lead ferroelectrics opens the door to the development of a science-based manufacturing technology that could revolutionize the industry.

Success would transform Sandia into one of the nationally and internationally recognized forerunners in the fabrication of texture-engineered ceramic materials, overcome the dependence on lead-based ferroelectric compositions, and provide an opportunity for improvements to existing technology used in the specialized ceramic components of our weapons systems.

Biotechnology

Studies of Signaling Domains in Model and Biological Membranes through Advanced Imaging Techniques

67010

A. R. Burns, K. Lidke

Project Purpose

Cellular membranes have complex lipid and protein structures that are laterally organized for optimized molecular recognition and signal transduction processes. Knowledge of nanometer-scale lateral organization and its function is of great importance in the analysis of receptor-based signaling, particularly signaling in response to toxins.

In model membranes, we study in detail the chemical and physical factors that result in lateral organization of lipids and lipid-mediated protein sequestration into signaling domains. We also map the location and follow the dynamic activity of specific membrane proteins involved in the immunological response of mast cells in collaboration with the University of New Mexico (UNM) Health Sciences Center.

These studies are enabled by development of new imaging methods that provide both high spatial resolution and sensitivity to dynamical processes. Our technical approach is to combine the high sensitivity and time resolution of fluorescence imaging with the high lateral resolution of atomic force microscopy (AFM). Simultaneous fluorescence and AFM imaging allows correlation of the distribution and dynamic activity of specific biomolecules via fluorescence labeling with complete topographic information of the membrane. We successfully used AFM to image topographic features in membranes with less than 1 nm lateral and vertical resolution. We also demonstrated our ability to detect fluorescence emission from single molecules.

Since most cellular signaling domains are smaller than the 200 nm diffraction limit imposed on fluorescence imaging, we will develop a fluorescence imaging technique that uses the highly localized excitation provided by the strong near-field enhancement at a metal-coated AFM tip. Overall, our unique imaging capabilities enable us to examine membrane structure and function with much greater detail than was previously possible and thus provide a better understanding of cellular signaling.

FY 2006 Accomplishments

We used combined AFM and fluorescence imaging techniques to analyze membrane organization in model membrane systems as well as biological membranes harvested from RBL-2H3 (rat basophilic leukemia) mast cells. In the multicomponent model membranes, we characterized partitioning of four fluorescence probes (headgroup- and tailgroup-labeled GM1 ganglioside probes, headgroup- and tailgroup-labeled phospholipid probes) between gel (saturated dipalmitoylphosphatidylcholine) and disordered fluid (unsaturated 1,2-dioleoyl-sn-glycero-3-phosphocholine) phospholipid domains. We mapped the domains with AFM topography and found partitioning of the probes between the gel and fluid domains to be a function of probe structure and how much it perturbed the packing of the host lipids.

In addition, we used fluorescence correlation spectroscopy to examine mobility of the labeled probes at specific sites in the model membranes. We saw over three orders of magnitude slower mobility for probes in the densely-packed gel domains relative to the fluid domains. For the GM1 probes, we observed large differences in mobility before and after binding toxin proteins.

In the biological membranes, we showed that specific receptors and membrane-associated signaling proteins labeled with fluorescent markers will localize in the membrane following activation of the immune response. Furthermore, the localization correlates with specific topographic features in the cytoplasmic leaflet mapped out with the AFM. We believe those features are invaginations of the membrane (clathrin-coated pits) that eventually lead to endocytosis of receptors.

Finally, we used quantum dots to label specific lipid components in both model and biological membranes. Quantum dots are very bright and have extremely large two-photon cross-sections. We observed two-photon fluorescence images of model membranes. These developments should allow us to implement near-field enhancement for sub-diffraction fluorescent resolution.

Significance

Innovative imaging technologies are critically needed to support future biotechnology research for Sandia and DOE. Recent requests for proposals by the DOE Office of Biological and Environmental Research (Genomes to Life), National Institutes of Health (National Institute of Biomedical Imaging and Bioengineering), and the Alliance for Cellular Signaling underline the importance of imaging in the characterization of biological systems.

Finally, we can have a significant impact on efforts to understand bioagent (toxin) activity at the molecular level and thus impact potential Department of Defense or Department of Homeland Security programs in this area.

Refereed Communications

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Other Communications

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Protein Microarrays for Biowarfare Agent Detection and Characterization

67012

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Project Purpose

The study focused primarily on developing customized protein microarrays to detect toxins and host-response to stimulation by toxins. As part of the work, we established custom protein microarray protocols, implemented a protein microarraying pipeline at Sandia, demonstrated detection of biologically relevant analytes including select agents, characterized the performance of the assay method, and identified shortcomings with the technology.

Establishment of a protein arraying capability at Sandia has been important both to this project as well as a resource to other ongoing projects at Sandia. Our team viewed this final year of the project as a prime opportunity to expand the scope and impact of the groundwork performed during the first two years of the project. As such, the purpose of the pipeline was, and continues to be, to enable rapid creation of custom microarrays for the study of interactions of low-concentration species and interactions of analytes in biologically complex samples (e.g., serum, saliva).

In addressing detection of toxins and biothreats, we developed an understanding of the targeted interactions, metrics for characterizing those interactions, and the optimal means to detect protein-protein as well as protein-antibody interactions. Before our work, a comprehensive investigation of these topics had not been reported in the literature – especially regarding detection of select agents. Biosensors relying on surface immobilization and immunosensing have been touted for years as ideal systems for biothreat detection and identification.

The purpose of our work in this area was to develop a multiplexed analytical means to fulfill both the detection and identification functions required. Development of this multiplexed capability, especially analysis of complex fluids, would be a step toward solving the problem of biothreat detection post-infection.

Sandia has been uniquely situated to conduct research on protein microarray development for biothreat sensing. Our capability to work with select agents allows Sandia to delve into the characterization and detection questions without being limited to working with select agent toxin surrogates. Based on the limited available public literature regarding these studies, our present work fills a niche that is important to national security concerns and provides empirical information not currently available to the research community.

FY 2006 Accomplishments

We focused on developing innovative assays for probing several facets of host-toxin interaction. Three primary interests defined our focus during this third project year: 1) enabling rapid methods to speed detection using small volumes of physiological fluids, 2) developing means to investigate host-response against a panel of possible toxic agents, and 3) refining the sensitivity and expanding the applicability of our assay format. The following list highlights our noteworthy technical contributions from FY 2006:

Photograftable Antibodies for Protein Microarrays

Grafting through solvated mobile polymer chains (e.g., acrylated antibodies in the presence of polyethylene glycol monoacrylate) allows rapid, ultraviolet patterning of oriented antibodies for higher sensitivity detection of low-concentration biothreat agents as well as antigens in complex biological fluids.

- Developed chemistries to enable photopatterning of antibodies on planar substrates and in microfluidic channels
- Analyzed compatibility of photopatterning methods with antibody and antigen immobilization
- Performed metrology studies of acrylate-modified surfaces to determine effectiveness of functionalization
- Characterized impact of acrylate-modified antibodies on affinity for target antigen

- Tested photopatterned antibody protocols on planar glass substrates and in microchannels fabricated from quartz and fused silica

Reverse-Phase Arrays for Measuring in vivo Response to Infection/Toxins
Reverse phase arrays, where the arrays consist of immobilized biological samples (e.g., serum), promise to enable profiling of low-abundance analytes and, consequently, studies of signal pathways. We focused on developing this powerful tool to use in readily collectable biological fluids such as saliva.

- Designed protocols for fabrication of "reverse-phase" arrays on a suite of substrates (epoxy, aldehyde, nitrocellulose membranes)
- Characterized effectiveness of patterning model biological sample matrix (saliva) on affinity of model antibody for target antigen (IL-1b, MMP-8)
- Developed high-sensitivity detection scheme involving chemical signal amplification to detect low-abundance analytes in biological sample
- Proposed new methods for preparing sample matrix for spotting

High-Specificity Sandwich Arrays for Presymptomatic Toxin Detection
Sandwich assays in conventional formats such as ELISAs (enzyme-linked immunosorbent assays) have proven to be exquisitely sensitive. We developed direct and competitive microarray formats in the previous two years of the project. Work in the final year centered on advancing the high-specificity sandwich format.

- Characterized substrate chemistries and linkage methods
- Tested signal amplification schemes for enhanced detection of binding
- Expanded the antigen panel beyond toxins to markers of inflammation important to measuring host response to toxin (cytokines, chemokines)
- Assessed levels of cross-reactivity for the expanded panel

Significance

Over the last three years, this research has transitioned from a high-risk endeavor to a consistent tool available to Sandia researchers. The methodologies proven during our study will have an impact on biosensor development, clinical diagnostics development (especially as related to detection of immune response to toxins), and studies concerning protein-protein interactions (especially as related to toxin pathways). Further, our team has introduced innovative biofabrication methods, including those that allow photopatterning of active biological molecules in micro- and nanosystems.

Defense and science are two of the four strategic goals of the Department of Energy. The core technologies developed and demonstrated as part of this project address defense innovation by significantly advancing the area of toxin and response-to-toxin sensing. Such studies and tools will potentially aid in diagnosis, possibly even early diagnosis, and protection of the US population and our defense forces world wide. The technologies developed as part of this research study also provide a general experimental science platform for characterizing protein-protein interactions and alternate detection moieties.

Refereed Communications

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Other Communications

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Interaction of Proteins with Lipid Films

67013
M. S. Kent, M. P. Sears, L. J. Frink, D. Y. Sasaki

Project Purpose

Diphtheria toxin (DT) contains separate domains for receptor-specific binding, translocation, and enzymatic activity. Following binding to cells, DT is taken up into endosome-like acidic compartments, where the translocation domains insert into the endosomal membranes and release the catalytic domain into the cytosol of the respective target cells. The processes by which the enzymes are translocated across the endosomal membrane are known to involve pH-induced conformational changes within (at least) the translocation domain; however, the molecular mechanisms are not yet understood.

We used neutron reflection to study pH dependent conformation changes of the nontoxic mutant CRM197 adsorbing to Langmuir monolayers of dipalmitoylphosphatidylglycerol in order to:

- Illucidate the mechanism of enzyme translocation
- Characterize and implement a tethered bilayer platform for investigating protein-membrane interactions using neutron reflection. A tethered bilayer is a lipid bilayer membrane that is immobilized to the substrate through a tethering lipid that contains a hydrophilic spacer underneath the bilayer to create a hydrated cushion.
- Use neutron reflection to determine the degree of completeness of the lipid bilayers and also the extent of hydration underneath the bilayer. High-quality, defect-free, hydrated tethered bilayers will enable us to study the structure of proteins associated with or inserted into lipid membranes.
- Study the adsorption of synthetic alanine-rich peptides to lipid monolayers by x-ray reflectivity and grazing incidence x-ray diffraction. The peptides contained histidine residues to drive adsorption to Langmuir monolayers of lipids with iminodiacetate headgroups loaded with Cu2+. The peptides were partially helical in solution at the temperature of the adsorption assays.
- Demonstrate control over the orientation of the peptides and also to determine the extent of conformational change upon binding as a function of the number of histidines (from 0 to 2) and the positioning of the histidines along the backbone.
- Explore the effects of alcohols on fluid lipid bilayers using a molecular theory with a coarse-grained model. Short-chain alcohols have significant effects on the physical properties of biological membranes. Changes in the membrane properties can lead to changes in the conformational states of intrinsic membrane proteins, thus leading to an indirect (nonspecific) mechanism for the modulation of protein behavior by alcohol adsorption into lipid membranes. Such indirect interactions are likely responsible for the role alcohols play in general anesthesia and in alcohol toxicity in bacterial and yeast cells.
- Use a molecular theory to explore the structure and energetics of assemblies of peptides embedded in lipid bilayers. Pores formed in biological membranes by the assembly of several (4-12) peptide fragments are a common motif in biological systems. Often the peptide units are amphipathic cationic-helices. The pores formed by these peptide assemblies are the basis for toxicity for a wide range of antimicrobial peptides and are part of the mechanism used by some toxins and viruses in penetrating cell membranes.

FY 2006 Accomplishments

The data for DT revealed a structural transition in the membrane-associated protein as a function of bulk DT concentration that also correlated with the change in the mode of segmental insertion. At 0.3 μM, the data indicated shallow insertion of segments into the membrane and a dimension normal to the membrane consistent with DT monomers. At 0.8 μM, the data indicated insertion of segments throughout the entire depth of the lipid film and a dimension of the protein layer normal to the membrane consistent with DT oligomers.

Solutions of CRM197 run on nondenaturing gels showed no evidence of oligomeric DT in solution over the range of DT concentration and pH of the neutron reflection measurements, indicating that oligomerization must occur upon adsorption.

We characterized a tethered bilayer system involving thiol tethers with polyethyleneglycol spacers and aliphatic lipid tails attached to gold surfaces. We deposited bilayers of dimyristoylphosphatidylglycerol using a rapid solvent precipitation method. Characterization with neutron reflection indicated nearly defect-free bilayers (detection limit approximately 2 percent D_2O) with a 15 Å hydrated layer containing 23 percent water underneath. We then used this bilayer system for further experiments with diphtheria toxin.

For the study of synthetic peptides, we observed that peptides containing two histidines with a spacing of 8 Å adsorbed to a final density and thickness consistent with densely packed-helices. With a spacing of 30 Å, we observed a much lower adsorbed amount and layer thickness. Furthermore, we observed striking differences in the interaction of the two types of peptide with lipid membranes by grazing incidence x-ray diffraction. Overall, the results suggested multiple-site binding and retention or increase of helical character upon binding for peptides with a spacing of 8 Å between histidines, but single-site binding and loss of helical character upon binding of peptides having a spacing of 30 Å between histidines. We also demonstrated control over the orientation of the peptides through placement of the histidines either in the center or on the end of the sequence.

In our work on alcohols in lipid membranes, trends predicted from the theory for the changes in area per lipid, alcohol concentration in the bilayer, and area compressibility modulus as a function of alcohol chain length and of the alcohol concentration follow those found experimentally. Using the theory to study the effect of alcohol on the lateral pressure profile across the membrane, we found that alcohol reduces the surface tensions at both the headgroup/solvent and headgroup/tailgroup interfaces, as well as the lateral pressures in the headgroup and tailgroup regions.

In the study of pore-forming peptide assemblies in lipid bilayers, we found both barrel-stave and toroidal pore morphologies for the lipids near the peptide assemblies but at different assembly sizes. The free energy of the assembly had a global free energy minimum for a solution with a membrane-spanning toroidal pore. A pairwise approximation to this free energy underpredicted the free energy minimum associated with the membrane-spanning pores.

Significance

The significance of the DT work is that it strongly suggests that oligomerization plays a role in the mechanism of enzyme translocation. Understanding this mechanism will facilitate the design of variants of these toxins for therapeutic purposes. Furthermore, based on structural similarities, aspects of the mechanisms may be common to a number of other proteins. In addition, the work with DT receptors should be seen as a step toward applying these methods to study botulinum toxin, a potential terrorist threat for which better post-exposure therapies are needed.

The significance of developing a high-quality, defect-free, hydrated tethered bilayer platform is that it will enable us to study the structure of proteins associated with or inserted into lipid membranes by neutron reflection. Structural information on membrane-associated proteins is desperately needed, as very few crystal structures of such proteins are available. Specific examples include the cellulosome, a large multienzyme protein complex used by some microbes to efficiently break down crystalline cellulose. Understanding the structure and assembly principles of this complex may facilitate the design of optimized cellulosomes and reduce the cost of producing ethanol from cellulosic biomass.

Another important complex that could be studied in this manner is the cellulose synthase rosette complex. Understanding its structure could facilitate the design of mutant forms to produce cellulose that is more easily broken down, again lowering the cost of producing ethanol from cellulosic biomass.

Another potential application is to study toll-like receptor complexes using these methods to determine the structures of TLR4 and MD2, and to study the association of lipopolysachharides with the TLR4/MD2 complex. Detailed knowledge of the TLR4/MD2 complex and its interactions with lipopolysachharides will enhance our understanding of pathogen recognition, provide insights into how pathogens evade the host immune system, and facilitate development of therapeutics designed to boost the immune response.

In our work with synthetic peptides, controlling the interface between lipid membranes and solutions of biological analytes is important for sensing, stimulating the formation of specific supramolecular structures useful for nanoscience applications, developing synthetic signaling systems, and for biophysical studies. Understanding the protein-membrane interaction and the dynamics of the adsorption process in greater detail will benefit many applications such as the localization of antibodies to substrates in biosensors in an orientation that exposes the binding site(s).

Our work on alcohols in membranes may help to explain fundamental limitations on alcohol tolerance of yeast or microbes used in biofuels production from cellulosic biomass. Our work on peptide assemblies in lipid bilayers could be seen as a first step toward more complex membrane protein structures, such as sugar transporters in yeast or microbes. Understanding these transporters could lead to engineered strains of yeast or bacteria that more efficiently take up five and six carbon sugars for conversion to ethanol.

Refereed Communications

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A.L. Frischknecht and L.J. Frink, "Effects of Alcohols on Lipid Bilayers from Molecular Theory," presented at Biophysical Society 50th Annual Meeting, Salt Lake City, UT, February 2006.

L.J. Frink, A.L. Frischknecht, and M.S. Kent, "Studying Proteins in Lipid Bilayers from Molecular Theory," presented at Biophysical Society 50th Annual Meeting, Salt Lake City, UT, February 2006.

New Technologies for Understanding Membrane Protein Recognition and Signaling

67014
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Project Purpose

Structural elucidation of membrane proteins is often difficult by standard techniques (e.g., x-ray crystallography and nuclear magnetic resonance) because of the hydrophobicity of the proteins, thus development of alternative strategies is paramount. Membrane proteins make up about 30 percent of our genome and are of profound importance in pharmacology and host-pathogen interactions. Understanding their properties is, therefore, of great importance for biodefense.

The purpose of this project is to demonstrate new technologies for measuring the structure, dynamics, and interactions of membrane proteins. Ideally, these methods are useful in the native living system (i.e., they don't require purified protein), are able to be used on small amounts of protein, and are useful for other types of structural and soluble proteins. The techniques we used are based on chemical cross-linking and mass spectrometry, particularly on the MS3D (mass spectrometry in three dimensions) technique, which combines chemical cross-linking, mass spectrometry, and theoretical modeling.

FY 2006 Accomplishments

We have invented a new technique for measuring structure and dynamics in proteins that we term SCRaM: sequential cross-linker rate measurements. In this technique, we measure the intensity of the product peaks from proteolytic digests of cross-linked proteins using mass spectrometry, and calculate ratios of peak intensities that can be related to cross-linking reaction rates.

By using a series of cross-linkers of different lengths, we can measure the parametric dependence of the rate on the cross-linker lengths. We also established a method for calculating these rate curves from first principles, based on the kinetic theory of macrocyclization reactions. We are using these techniques to fit distances to data measured from Type-II polyproline helices, alpha-helical peptides, and the membrane protein bovine rhodopsin.

Significance

The SCRaM technique is, in principle, suitable for quantitatively measuring structural and dynamic properties in membrane proteins in a manner similar to fluorescence methods such as fluorescence resonance energy transfer (FRET). However, it can yield distance information and dynamical information on any protein containing, for example, one or more reactive cysteine residues, eliminating the need for double mutations and labeling. And because proteins are purified after being cross-linked but prior to analysis, they can be analyzed in their native environment.

Given the enormous number of biological applications of FRET, and the potential advantages of SCRaM over FRET, we believe that SCRaM may have a broad utility in biological sciences when structural and conformation characterization of proteins and protein or other macromolecular complexes need to be understood.

Refereed Communications

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Integrated Genome-Based Identification of Biological Agent Proteins: A Microfluidic Module for Nanosequencing of Proteins and Peptides

79745

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Project Purpose

The purpose of this project is to develop a microfluidic protein nanosequencing device (NSD) to determine terminal amino acid sequences from nanoliter volumes of protein solution. The NSD is a near-reagentless alternative to DNA- and affinity-based agent identification. Orthogonal detection is critical in cases where there are few or no stable DNA signatures (i.e., viruses, toxins). As has been shown by research using mass spectrometry (MS)-based pathogen detectors and existing protein databases, protein sequence information enables species-specific identification of threat organisms with high surety. Like MS, the NSD could also rapidly characterize unexpected proteins from engineered organisms and ultimately serve as a miniature, highly parallel replacement for MS.

The nanosequencer will use enzymatic sequencing methods to serially liberate and identify terminal amino acids from the protein. Short (4-5 amino acids) sequences are often sufficient to determine the exact identity of a protein. Characterization of a small number of proteins from a given organism will permit its certain identification. The device, which will consist of a microreactor, an amino acid separator, and an analyzer, will identify the amino acids using either microseparations coupled with laser-induced fluorescence or antibody detection. Integration of these components into a nanosequencer requires unique Sandia technologies.

The project would produce valuable intellectual property in that the device has the potential to replace mass spectrometry for bioagent detection and commercial applications (proteomics, drug development). The device is a logical extension of protein-based identification systems. Once demonstrated it would serve as the foundation for next-generation microfluidic detection platforms for the Department of Homeland Security (DHS) or Department of Defense.

FY 2006 Accomplishments

We made significant progress toward the development of immobilized enzymatic reactors for peptide and protein sequencing. We had previously demonstrated that using a mixed bed reactor consisting of immobilized carboxypeptidases B and Y (CPBY) resulted in more efficient C-terminal cleavage of peptides than using either carboxypeptidase B or carboxypeptidase Y alone.

Our focus this year was to optimize conditions for sequencing of whole proteins. The CPBY reactor cleaves three C-terminal amino acids from the low molecular weight protein ubiquitin (approximately 8 kDa) within five minutes. We observed no carboxypeptidase digestion products with higher molecular-weight test proteins, which we attributed to the inaccessibility of the C-terminal. We therefore developed an alternative strategy for a microfluidic protein sequencing system that is based on the integration of discrete trypsin, anhydrotrypsin (AHT), and CPBY microreactors coupled to a detector module using low dead-volume capillary-to-capillary connectors.

In this design, protein is delivered under pressure to the trypsin reactor and the C-terminal tryptic peptide is isolated using the AHT affinity column and sequenced with CPBY. We developed procedures for immobilizing trypsin and AHT onto silica beads, which were packed and retained into robust microliter cartridges. We coupled these enzymatic reactors using low dead-volume interconnects and reproducible tryptic digest, and demonstrated subsequent C-terminal isolation of casein (23 kDa) and BSA (bovine serum albumin, approximately 64 kDa).

Significance

The microfluidic-based protein sequencing system is intended to support the Department of Energy's national security mission by offering a unique third bioagent detection option to DNA methods and antibody/affinity-based methods, since it is clear that situations will arise when these methods will fail. This technology could possibly form the foundation for a successor to the MicroChemLab™ and BioBriefcase programs now funded by the DHS. It will build Sandia's base in biotechnology and microsystems science and create a fundamentally new technology. We also developed a new method for biological sample analysis coupled to MS. National Institutes of Health opportunities might arise from this work.

Refereed Communications

M.J. Ayson, R.B. Jacobsen, J.S. Schoeniger, and R. Shediac, "Characterization of Immobilized Carboxypeptidase Reactors for Protein Sequencing," in *Proceedings of the 54th ASMS Conference*, May 2006, DVD.

Tools for Characterizing Membrane Rafts and Toxin Interactions

79746
T. M. Alam, A. Costello, M. J. Stevens

Project Purpose

The formation of rafts (or lateral reorganization) in membranes plays an important role in cell function. Understanding these rafts provides input for the rational development of physically-based system-biology models describing bioagent and toxin interactions with membranes. Numerous experimental and computational methods are currently used to explore membrane-protein interactions, including ongoing atomic force microscopy and fluorescence resonance energy transfer research at Sandia, along with computational dynamics simulations. Still, many questions concerning the biochemistry and biophysics of these interactions remain unanswered.

The purpose of this project is to develop nuclear magnetic resonance (NMR) spectroscopy tools that allow for the experimental measurement of the domain size and chemical composition of biomembrane "rafts" or microdomains. These NMR tools will then be used to analyze toxin-membrane interactions and the impact of rafts on the resulting biochemical activity. This research involves the development of NMR experimental spin-diffusion, nuclei specific editing, and dipolar recoupled radio-frequency pulse sequences that can detect and measure membrane raft domain size and dynamics, as well as probe toxin binding at the membrane surface.

FY 2006 Accomplishments

The FY 2006 research focused on the measurement of domain (raft) size in biomembranes. Our research resulted in numerous presentations, three publications, and several manuscripts in preparation. The work has been well received at professional meetings, and there has been significant interest in our ability to obtain information about different membrane constituents and how they interact without resorting to chemical tags of perturbing labels. In addition, we made significant progress on the year's milestones:

Milestone 1: Measure Raft Domain Size in Model Membranes versus Temperature and Composition

We completed the initial NMR studies on the dioleonylphosphocholine (DOPC)/sphingomyelin (SM)/cholesterol (CHOL) model membrane system. The DOPC/SM/CHOL system represents the canonical raft-forming model system. We completed a ^{31}P magic angle spinning (MAS) NMR study of the DOPC/SM/CHOL system for different concentrations and as a function of temperature and obtained several key results:

- These experimental results demonstrate that the conceptual description of rafts, or domains, needs to be extended to include details about the phosphate headgroup environment and dynamics when describing rafts.
- These 31P NMR experiments also allow a limiting value on the raft domain size in the gel-to-liquid-ordered coexisting region to be measured, while for the liquid-ordered-liquid-disordered region, the 31P NMR results could not provide a direct measure of the domain size.

Milestone 2: Compare NMR Experimental Raft-Size Results with Other Results and Simulations

- We developed a 1H-1H mediated spin diffusion experiment that allows us to measure the domain size in the DOPC/SM/CHOL model membrane system. This method overcomes the sensitivity limitation of the 31P-31P experiments.
- To address the simulation component of this milestone, we initiated molecular dynamics (MD) simulations for the sphingomyelin-containing membranes and finite element analysis of spin diffusion events. We are pursuing the MD simulations to address a unique set of water contacts observed in the two-dimensional 1H-31P NMR correlation experiments. The domain size and time scale of these membrane bounds

waters are intriguing and raise the question of how important these water-lipid interactions are in governing raft or domain formation. We are comparing the MD simulation data to the NMR measurements of pure SM membranes. Once the SM force fields have been developed, we will extend these MD simulations to include the DOPC/SM/CHOL system for which we have NMR data on domain size.

Significance

The present working description of rafts has concentrated on the ordering of the acyl chain (liquid-ordered versus liquid-disordered) with no real insight into the headgroup environment. The atomic level results obtained from the NMR in this project demonstrate that this working paradigm needs to be extended to include a molecular understanding of the entire lipid molecule. We argue that this headgroup region is also important, since interactions with many cellular components (proteins, toxins, viruses) will occur within this region, and should be included in any future description of membrane rafts.

The research in this project also provides a basis for future Sandia work on membranes and may be used to address the new National Institutes of Health lipid roadmap.

Refereed Communications

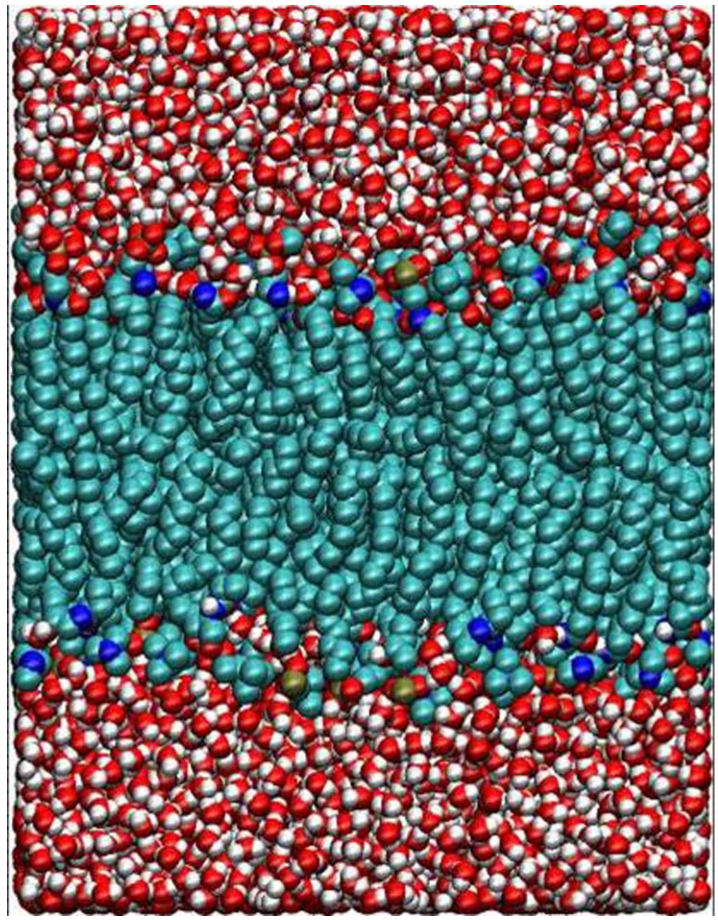
G.P. Holland, S.K. McIntyre, and T.M. Alam, "Distinguishing Individual Lipid Headgroup Mobility and Phase Transitions in Raft Forming Lipid Mixtures with ^{31}P MAS NMR," *Biophysical Journal*, vol. 90, pp. 4248-4260, 2006.

G.P. Holland and T.M. Alam, "Multidimensional ^{1}H-^{13}C HETCOR and FSLG-HETCOR NMR Study of Sphingomyelin Bilayers Containing Cholesterol in the Gel and Liquid Crystalline States," *Journal of Magnetic Resonance*, vol. 181, pp. 316-326, 2006.

T.M. Alam and G.P. Holland, "^{1}H-^{13}C INEPT MAS NMR Correlation Experiments with ^{1}H-^{1}H Mediated Magnetization Exchange to Probe Organization in Lipid Biomembranes," *Journal of Magnetic Resonance*, vol. 180, pp. 210-221, 2006.

Other Communications

T.M. Alam and G.P. Holland, "2D ^{1}H-^{31}P Dipolar HETCOR NMR Probe of Molecular Contacts in Sphingomyelin Bilayers," presented at 50th Annual Biophysical Society Meeting, Salt Lake City, UT, 2006.



Molecular dynamics simulation of sphingomyelin

Integrated Nanosystems for Monitoring Cell-Signaling Proteins

79747

B. C. Bunker, R. Rebeil, K. R. Zavadil, W. G. Yelton, G. D. Bachand, M. Farrow, M. Okandan

Project Purpose

The objective of this project is to develop an integrated microfluidics platform that can be used for the real-time detection of proteins in the cell-signaling cascade that determines how the human immune system responds to pathogens. A fundamental understanding of human pathogenesis is critical to countering threats to national security posed by new pathogens that arise from natural selection or are deliberately engineered.

This project involves the development of sensor capabilities that will enable us to monitor the populations of cell-signaling proteins in real time, forming the basis for enduring partnerships in which Sandia's expertise in microsystems will eventually be coupled to strong molecular biology groups at other institutions, including Los Alamos National Laboratory, Southwestern Medical Center (University of Texas, Dallas), and the National BioContainment Laboratories (University of Texas Medical Branch, Galveston). Such partnerships will establish Sandia as a leader in the use of microsystems and nanotechnology to solve problems of interest to national security and human health.

Major components of the project include:

Protein Capture Monolayers

We are developing surfaces that can be electrochemically program-med with antibodies, allowing for highly selective adsorption of specific proteins on top of an underlying sensor array.

Sensor Elements

We are developing porous films that can function as ultracapacitors or electrochemical sensors. The currents generated in such films versus applied voltage are extremely sensitive to the presence of adsorbed proteins via a pore-blocking mechanism.

Integrated Microfluidics

We are developing fluidic devices that can immobilize and sustain individual cells, provide nutrients and toxins to the cells, and incorporate the sensors described above to detect cytokine production in real time.

Biological Testing

We are testing the integrated sensor platform with white blood cells to detect the production of cytokines such as interleukins (e.g., IL-2).

FY 2006 Accomplishments

Programmable Surfaces

We functionalized ferrocene functionalized with an antibody (rabbit IgG) and showed that cyclodextrin surfaces reversibly adsorb and desorb this antibody by reducing and oxidizing the ferrocene, respectively. We also demonstrated surface programming by observing the capture and release of quantum dots under a fluorescence microscope. These demonstrations confirm that we should be able to program surfaces with antibodies specific for capturing specific cell signaling proteins.

Ultracapacitor Sensors

We replaced mesoporous silica supports, initially used as ultracapacitors, with controlled-porosity anodized aluminum films. By varying anodization conditions, both monodisperse and bimodal pore distributions ranging in size from 5-1000 nm can be prepared. As both the dynamic range and sensitivity of the ultracapacitor for sensing proteins is dependent on pore blockage, the ability to control pore sizes relative to protein sizes will be critical for optimizing sensor performance.

Microfluidic Platforms

We fabricated and are testing a platform to capture single cells, support cellular functions, stimulate cells, sample proteins, and incorporate protein sensors.

Preliminary results indicate that the fluidic platform is compatible with white blood cells (macrophages) and that the patch clamp in the system controls the capture and release of single cells.

Biocompatibility Studies
We performed our first set of experiments to determine whether platform and sensor components are susceptible to biofouling (irreversible protein adsorption) and if living cells remain viable when in contact with sensor components. Experiments with the protein albumin suggest that biofouling is not a critical issue. Experiments with three cell types of interest (epithelial cells, macrophages, and monocytes) suggest that while most cells are "happy" in our system, selected cell-surface interactions can lead to cell activation and apoptosis (programmed cell death).

Significance

The overarching goal of this project is to develop an integrated "on-chip" laboratory that can detect the production of small quantities of proteins in single cells such as human white blood cells. To date, the individual components of such a system have been developed and tested, each of which is significant in its own right. The electrochemically-switchable films represent a new way of programming surfaces to grab and release species from solution, impacting our ability to perform functions in microfluidic systems such as preconcentrating, sorting, separating, and sensing species such as proteins, as well as assembling and reconfiguring nanomaterials.

The ultracapacitor sensors have the potential to revolutionize on-chip sensing of proteins. Small sensor electrodes have the potential to analyze as few as 10 protein molecules, allowing scientists to study processes occurring in single cells. The microfluidic system developed in this project provides the on-chip laboratory that allows scientists to capture single cells, expose the cells to different environments or stimuli, and monitor how the cells respond.

This system has already been transferred to the Microscale Immune Study Laboratory Grand Challenge LDRD project for monitoring cell responses. Once the films, capacitors, and microfluidics are integrated together in the third year of the project, the net system should be capable of providing a unique on-chip laboratory for studying the cell-signaling cascade that is critical to understanding the human immune response, as well as providing early warning of human exposures to biothreats for homeland defense.

Refereed Communications

M.J. Farrow, K.R. Zavadil, W.G. Yelton, and B.C. Bunker, "Electrochemically Switchable b-Cyclodextrin SAMs for Use in Biomicrofluidic Devices," presented at Pacifichem, Honolulu, HI, December 2005.

M.J. Farrow, K.R. Zavadil, D.L. Pile, W.G. Yelton, and B.C. Bunker, "Electrochemically Programmable Bioactive Interfaces at b-Cyclodextrin SAMS," presented at ACS National Meeting, Atlanta, GA, March 2006.

M.J. Farrow, K.R. Zavadil, D.L. Pile, W.G. Yelton, and B.C. Bunker, "Reversible Patterning of Antibodies in b-Cyclodextrin SAMs," presented at MRS Spring Meeting, San Francisco, CA, April 2006.

Other Communications

B.C. Bunker, "Active Interfaces for Integrated Biosystems," presented (invited) at Wright Patterson Air Force Research Lab, Dayton, OH, June 2006.

B.C. Bunker, "Active Interfaces for Manipulating and Exploiting Biomolecules," presented (invited) at Max-Planck Ringberg Symposium, Bioinspired Synthesis and Materials, Tegernsee, Germany, October 2006.

M. Okandan, "Microsystems for Characterization of Biological Systems," presented (invited) at BioMEMS Gordan Research Conference, New London, CT, June 2006.

DNA-Based Intelligent Microsensors for Genetically Modified Organisms (GMO)

79749
E. E. May, S. M. Brozik, P. L. Dolan, P. S. Crozier

Project Purpose

The ability to discriminate nucleic acid sequences is necessary for a wide variety of applications: high-throughput screening, distinguishing genetically modified organisms (GMOs), molecular computing, differentiating biological markers, fingerprinting a specific sensor response for complex systems, biometrics, and so on. Current DNA-based discrimination or sensor systems are simple, relying heavily on computational post-processing of noisy data for detection. Next-generation sensor-dependant technologies require intelligent DNA microsensors that are able to perform complex detection tasks such as systematic identification of GMOs in the presence of nonlethal agents.

We propose to combine two prevailing technologies, deoxyribozyme computation and catalytic molecular beacons, to design intelligent systems that upon discriminating specific sequences provide gate output oligonucleotides for parallel processing of recognition events. Our objectives are to:

- Develop capabilities needed for creating next-generation, intelligent DNA-based microsensors
- Construct a system that demonstrates intelligent sensing capabilities by identifying pathogens maliciously modified to bypass current detection schemes.

FY 2006 Accomplishments

The goal for FY 2006 was to demonstrate the sensors' ability to identify and detect mutations in DNA targets. Goals and milestones centered on developing a quantitative understanding of mutations on the output fluorescence of deoxyribozyme gates. We successfully investigated gate immobilization methods, developed multitarget gates, developed algorithms for classification of output fluorescence, and developed *in silico* methods for predicting fluorescence activity of various input/gate combinations. We have completed the following milestones:

Investigate immobilization schemes of the molecular beacons and show feasibility of patterning ribozymes in a manner conducive to Boolean calculations

- We investigated whether the YESiA (E6) deoxyribozyme/catalytic molecular beacon remains active after immobilization on a solid substrate. In order for the deoxyribozyme gates to remain active, they must be able to fold into their functional three-dimensional conformation.
- We demonstrated that biotin-labeled YESiA (E6) deoxyribozyme molecular beacons are able to fold properly and remain active. The immobilized YES gates demonstrated catalytic activity, as measured by increased fluorescence emission, albeit lower than YES gates in solution.

Develop and simulate algorithms for multiple input Boolean networks able to detect the simultaneous presence of multiple target

- In FY 2005 we developed a Xyce-based framework for simulating deoxyribozyme algorithms. Our focus in FY 2006 was to develop quantitative methods for both post-sensor classification and approaches for coupling classification and sensing events.
- We developed two classification approaches: a Bayesian classifier and a coding theory method. We found that, based on the fluorescence output, the Bayesian classifier correctly classifies the location of the single-nucleotide polymorphism (SNP) 71.1 percent of the time for a two-class (upstream/downstream) system and 62.2 percent of the time for a three-class (5', middle, 3') system. The coding theory classification method produced a binary code used to calculate syndromes for each SNP sequence. NonSNP input produced an all-zero syndrome, which in the coding theoretic sense indicates the absence of errors. Sequences with SNPs near the middle through 3' ends produced unique syndrome vectors that potentially can be used to classify mutations.

Demonstrate complex functionalities, including the detection of two different targets (ribozyme output should indicate whether one, two, or none of the targets are present)

- We demonstrated functionality of a 2-input deoxyribozyme gate (Normal iA_AND_iB) and enhanced activity by modification of the original gate sequence. The iA_AND_iB gate becomes active only when two complementary input sequences bind to each of its 15-base loop sequences. When only one input sequence bound to its respective loop, fluorescence output decreased significantly, nearly matching that of samples containing gate and fluorescent substrate without input DNA.

Complete molecular simulation of molecular beacon mismatch energetics

- We calculated binding affinities and made computational predictions of fluorescence, based on established correlation.
- We wrote new modules for LAMMPS to allow users to perform alchemical changes, rerun trajectories, and compute interaction energies. These new LAMMPS modules facilitate computation of free-energy changes due to nucleic acid mutations. SNP hybridization free-energies and ribozyme fluorescences can then be estimated from the molecular simulation data.

Significance

Hybridization-based target recognition and discrimination is central to the operation of nucleic acid microsensor systems. Therefore, developing a quantitative correlation between mis-hybridization events and sensor output is critical to the accurate interpretation of results. Additionally, knowledge of such correlation can be used to design intelligent sensor systems that incorporate mis-hybridization noise into system design.

If successful, this work will lead to development of intelligent sensors that can distinguish mismatches within DNA sequences. This can impact detection of genetically modified organisms, as well as bioremediation and bioterrorism defense, of interest to DOE and the Department of Homeland Security. It can also aid in the development of high-throughput DNA screening and encourage the development of DNA-based biometric devices.

Refereed Communications

E.E. May, P.L. Dolan, P.S. Crozier, and S.M. Brozik, "Syndrome-Based Discrimination of Single Nucleotide Polymorphisms," to be published in *Proceedings of the 28th IEEE EMBS 2006 International Conference,* August 2006.

S.M. Brozik, P.S. Crozier, P.L. Dolan, and E.E. May, "Quantitative Assessment of SNP Discrimination for Computational Molecular Beacons," in *Proceedings of the 2nd ASM - IEEE EMBS Conference on Bio-, Micro- and Nanosystems*, p. 35, January 2006.

Other Communications

S.M. Brozik, P.S. Crozier, P.L. Dolan, and E.E. May, "Quantitative Assessment of SNP Discrimination for Computational Molecular Beacons," presented at the 2nd Annual New Mexico Bioinformatics Symposium, Santa Fe, NM, March 2006.

Virulence Membrane Protein Organization and Complex Formation in *Francisella novicida*

93498
T. W. Lane, J. A. Timlin, J. N. Kaiser, M. B. Sinclair, L. Nieman

Project Purpose

This project addresses self-organization and complex formation among virulence proteins in *Francisella novicida*, a Risk Group 2 (RG2) organism related to the select agent *Francisella tularensis*. The proteins encoded by the pathogenicity island are the only major virulence factors in *Francisella*. These proteins form a multicomponent, integral membrane protein complex, which is postulated to be involved in membrane surface interactions between host and pathogen and transport of proteins through cell membranes and into the host cell cytoplasm. The formation of the virulence protein complex is absolutely required to cause disease and thus can serve as a detection marker and therapeutic target.

In this project we applied Sandia's unique capabilities in hyperspectral imaging and structural analysis to characterize the protein-protein interactions involved in virulence protein complex formation. We interrogated live cells with single and multiple genetically tagged virulence proteins via time-resolved fluorescence resonance energy transfer (FRET) on Sandia's hyperspectral microscope and elucidated how these interactions mediate pathogenesis. The ability to monitor multiple tags simultaneously in the pathogen and/or the host offers a view of host-pathogen protein interactions not available with any other technique.

We performed nearest-neighbor analysis of protein-protein interactions by cross-linking virulence protein complexes with linkers of different arm length followed by mass spectroscopy. Coupling the structural and functional information obtained in this project would facilitate the development of methods to short-circuit pathogenic bacteria. If successful, this work will provide a better understanding of mechanisms of infection and may generate follow-on funding from the National Institutes of Health (NIH) and biodefense programs.

FY 2006 Accomplishments

We completed the following milestones in FY 2006:

- Collaborated with Dr. Karl Klose (University of Texas, San Antonio) to image the movement of *Francisella* virulence proteins from the pathogen into the cytoplasm of the macrophage.
- Acquired most of the available *Francisella* vectors and plasmids encoding the new-generation fluorescent proteins.
- Recombined the existing *Francisella* plasmids with the fluorescent protein plasmids, creating a new generation of vectors that will offer a greater choice in the types of fluorescent and affinity-tagged fusions that we will be able to create in *Francisella*.
- Cloned the virulence genes, encoding the IglAB and part of the PdpABCD virulence protein complexes from *Francisella novicida*.
- Created strains of *Francisella novicida* containing fluorescently tagged constructs to the virulence protein AcpA and the control protein GroE and have imaged them with the hyperspectral scanner.
- Developed the protocol for the *Francisella* infection of macrophages, which will enable us to assay fusion protein constructs for proper function.
- Initiated a task to generate FRET pairs of the IglA and IglB virulence proteins.
- Use the fusions to track expression and localization of the proteins in *Francisella novicida* under conditions of simulated and actual infection of macrophages, which will lay the groundwork for the FRET analysis of the IglAB complex.
- Generate fusions for the FRET analysis of the PdpABCD complex.
- Developed the technical infrastructure to handle RG2 organisms in the Hyperspectral Imaging Laboratory.
- Develop a protocol for transfer of agents and seek the appropriate approvals and permits.

Significance

Identifing and characterizing genetic and proteomic targets is critical to the Sandia's mission of developing technologies for detecting and responding to biological and chemical attacks. This research benefits that mission by characterizing a pathogen-specific membrane protein complex that could be used for detection and identification. In doing so, we advance our understanding of an important biological process relevant to *Francisella tularensis* pathogenesis.

It is thought that sequence variations within the virulence island (and the proteins it encodes) are responsible for the range of infectivity that is seen among the different subspecies and strains of *Francisella*. A fuller understanding of the multiprotein structures that are responsible for pathogenesis will facilitate the process of analyzing the risk associated with different strains.

This project is designed to be the first phase of an open-ended research program and will provide results that answer important fundamental questions in microbiology. If successful, our work will:

- Establish a track record in the analysis of *Francisella novicida* resulting in high-profile publications and extramural funding.
- Provide preliminary results that may be used to respond to a NIH/National Institute of Allergy and Infectious Diseases biodefense call.
- Support future directions of both the Biotechnology and the Homeland Security investment areas at Sandia.
- Enhance our ability to characterize multisubunit membrane protein complexes that have important implications for biodefense.
- Provide an understanding of the structure and function of this virulence complex, which will facilitate risk analysis efforts to understand the level of virulence posed by different strains.
- Benefit Department of Defense and Department of Homeland Security efforts to generate proteomic signatures of bioweapons strains and to develop ligands for the detection of agent-specific surface protein domains.

Cell Modeling with Heterogeneous, Dynamic Cell Membranes

93499

L. J. Frink, H. D. Jones, M. B. Sinclair, D. M. Haaland, S. J. Plimpton, S. M. Brozik

Project Purpose

This project is a collaborative effort between experimental and computational scientists to develop new methods for studying cells as they interact with antagonist peptides. We are focusing on *Escherichia coli* and several antimicrobial peptides (AMPs), including magainin-2, buforin-II, and arenicin-1. Magainin-2 and buforin-II are thought to have quite different mechanisms with respect to cell toxicity. Magainin-2 is a pore-forming peptide that kills the cell by increasing the porosity of cell membranes. In contrast, buforin-II disrupts the DNA machinary of the cell. The mode of action of arenicin-1 is unknown.

Our experiments seek to identify and quantify the action of different AMPs using imaging methods. From the perspective of the experimental biology, the work is novel in applying hyperspectral imaging methods to cell-AMP interactions. From the computational perspective, we propose to build new higher-fidelity membrane models for ChemCell (cell modeling software developed at Sandia) using a multiscale approach to provide molecular-level input to the cell model via an intermediate lattice model for the cell membrane. In addition, we propose to extend ChemCell in the direction of a cell life cycle model in order to model AMPs that kill cells via interaction with DNA.

FY 2006 Accomplishments

We made significant accomplishments in both the experimental and computational parts of this project.

The experimental effort has progressed further than expected this year. We have:

- Identified a collection of AMPs, magainin-1, buforin-II, and arenicin-1 to investigate.
- Completed initial assays to determine the potency of both unlabeled and fluorescently labeled AMPs against *E. coli*, our model bacteria.
- Performed imaging experiments on porous silica beads coated with labeled lipid bilayers.
- Demonstrated that we can distinguish between bilayers and AMPs.
- Observed fluorescence resonance energy transfer (FRET) occurring for magainin-1 and buforin-II, and in both cases the AMPs penetrate the membrane.

In the computational part of the project, we have:

- Completed a variety of tasks to augment ChemCell with features that will enable a coupling with a mesoscale membrane model and enable testing the code on prototypical cell signaling problems.
- Tested the new features on a chemotaxis signaling model in *E. coli*.
- Presented an overview of our cell modeling capabilities in invited talks at two recent conferences: the Biophysical Society Annual Meeting and the Society for Industrial and Applied Mathematics Conference on Parallel Processing for Scientific Computing.

Molecular modeling on AMPs interacting with lipid bilayers has focused on the thermodynamics of pore-forming assemblies of alpha-helix forming peptides. While the models have been quite generic, we have demonstrated complex lipid structures in the vicinity of the assemblies, computed free energies of pore formation, identified an underlying first-order phase transition, and investigated variations in peptide chemistry.

Significance

The significance of the experimental work is that it is the first step in developing capabilities for direct hyperspectral-based imaging of cells interacting with antagonists with spatial and temporal resolution. These studies when completed will provide a platform to consider a broader suite of both antagonists and cell lines. Direct imaging of cell-antagonist interactions will elucidate modes of action for antagonists in various situations.

The significance of the cell modeling work completed this year is that systems with spatially complex membranes have been treated for the first time with ChemCell, an emerging technology at Sandia that is unique in modeling cells with spatial resolution. Furthermore, molecular modeling work has identified a thermodynamic phase transition (or switch) associated with the formation of membrane-spanning pores in lipid bilayers. This is the mode of action of magainin-2 (one of the antimicrobial peptide targets of the project). The existence of this phase transition provides a basis for the development of synthetic nanosystems that can use this switch to alter their properties.

Refereed Communications

L.J. Frink, A. Frischknecht, and M. Kent, "Studying Proteins in Lipid Bilayers Using Molecular Theory," presented at Biophysical Society, Salt Lake City, UT, February 2006.

S.J. Plimpton, "Modeling Protein Networks in Cells via Simulations of Diffusing Reactive Particles," presented at Biophysical Society, Salt Lake City, UT, February 2006.

L.J. Frink, M.G. Martin, A.G. Salinger, and M.A. Heroux, "High-Performance Computing for the Application of Molecular Theories to Biological Systems," in *Proceedings of the SciDAC 2006, Scientific Discovery Through Advanced Computing*, p. 304, June 2006.

S.J. Plimpton, "Biological Cell Modeling: Progress and Challenges," presented at SIAM Parallel Processing for Scientific Computing, San Francisco, CA, February 2006.

S.J. Plimpton, "Biological Cell Modeling via Simulations of Diffusing Reactive Particles," presented at DOD HPC Users Group Conference, Denver, CO, June 2006.

Other Communications

L.J. Frink, "A Thermodynamic Switch Controlling Membrane-Spanning Pores in Biological Membranes," submitted to *Nature*.

Membrane Analysis of the Plague Bacterium *Yersinia pestis* during Flea to Mammalian Host Adaptation

93500
R. Rebeil, K. K. Crown, L. N. Brewer, J. A. Ohlhausen, M. Z. Hadi, S. P. Gaucher, B. Ricken

Project Purpose

Yersinia pestis, the causative agent of plague, is classified as both a potential agent of bioterrorism and as a re-emerging threat to world public health. The mortality rate displayed in both the bubonic and pneumonic forms, 30-40 and >95 percent respectively, is one of the highest among all class A pathogens. In addition, unlike most select agents that only have one route of dissemination, *Y. pestis* can be deployed by terrorists via aerosols and via its natural flea vector.

Due to its deadly nature, *Y. pestis* was one of the first microbial agents to be weaponized and was successfully deployed by the Japanese using infected vectors (fleas) against human populations during World War II. In addition, plans were under way before the end of the war to release plague and typhus onto large West coast cities, such as San Francisco, using infected vectors.

Because plague displays one of the highest lethality rates of all the select agents when spread via aerosols, most biodefense research efforts to date have concentrated on this route of exposure. Yet release of this agent in aerosol form would likely require significant physical resources and technical skill, which may not be possessed by many adversaries, such as terrorists. Thus, distribution via its natural vector is also a significant biodefense concern. Because of its lethality and varied potential dissemination routes, *Y. pestis* is a high priority for study among the select agents.

In order to advance Sandia's efforts in biodefense and enhance its reputation in the biology research field, this one-year study was undertaken on membrane protein markers in *Y. pestis* that can be exploited for detection and identification in the case of an intentional release of plague. The study was designed to also identify useful markers that can be used to determine whether the bacteria are of natural or artificial origin (forensic applications).

To date, many proteomics projects have been hampered or their usefulness called into question because of poor understanding of the physiology of the microorganisms being studied. What differentiates this study from other similar proteomics efforts is the study concentrated on markers present in the bacterial membrane, which are more relevant for developing detection markers, and the range of growth conditions used was more extensive and biologically relevant than other similar studies as it takes into account the complex physiology of *Y. pestis*. For this project, we used the nonvirulent *Y. pestis* strain KIM6+.

FY 2006 Accomplishments

Growth conditions:

- We grew nonvirulent *Y. pestis* KIM6+ at 21, 28, and 37 degrees C at mid-logarithmic and stationary phases of growth.
- We grew nonvirulent *Y. pestis* KIM6+ in a novel in vitro media formulation that generates bacterial aggregates that highly resemble those generated by *Y. pestis* in vivo. The new media formulation is permitting the team to study factors involved in transmission.
- We isolated inner and outer membranes from cells growing in artificial clumps for analysis, which will yield important information on outer membrane physiology during the course of disease transmission.
- We grew cells in human and fetal bovine serum to understand bacterial physiology when the organism is growing in the circulation of mammalian hosts.

Proteomics analyses:

- We isolated inner and outer membranes from nonvirulent *Y. pestis* KIM6+, growing in all

different conditions used, and using sucrose density gradients. The initial goal was to isolate whole membranes.
- We solubilized, quantitated, and analyzed the membrane proteins using one-dimensional and two-dimensional gel electrophoresis techniques.
- We identified common protein bands that can potentially be used to effectively detect *Y. pestis* without false negatives.
- We isolated the most abundant proteins from gel slices for identification using mass spectrometry techniques.

Other analyses and data:
- We established a collaboration with B. Joseph Hinnebusch (National Institutes of Health), who is providing samples of flea-gut contents for analyses at Sandia.
- We used x-ray photoelectron spectroscopy to study the chemical composition of flea blood meals to determine if nutrient concentrations in the flea vector may contribute to the behavior observed inside the flea.
- We obtained carbon-nitrogen, carbon-oxygen, and oxygen-nitrogen ratios for mouse blood, flea blood meal one hour after ingestion, and flea blood meal 48 hours after ingestion.
- We identified major differences in the carbon-nitrogen and oxygen-nitrogen ratios in flea blood meals 48 hours after ingestion, indicating a dramatic shift in nutrient contents that may be important in the transmission of plague.

To our knowledge, this is the first such analysis of an important disease-spreading insect and lays the foundation for further analyses and collaborations with other major laboratories.

Significance

Through our research we have uncovered new research tools that can be exploited for further studies of disease-causing organisms. The novel in vitro growth system will permit the application of various genetic and molecular techniques to explore how bacterial *Y. pestis* factors contribute to transmission.

In addition, we have begun to apply techniques normally used in surface chemistry analysis, such as x-ray photoelectron spectroscopy and time-of-flight secondary ion mass spectrometry, to look at insect physiology and its role in disease transmission. This is highly relevant since many bioterrorism agents, as well as emergent and re-emergent diseases, are transmitted by insects.

Shotgun Protein Sequencing

93501
J. M. Faulon, Z. Zhang, A. K. Singh, S. P. Gaucher

Project Purpose

Gene sequence information alone does not provide a complete and accurate picture of a protein final sequence and state of activity. While current estimates put the number of human genes at about 30,000, the number of proteins is undetermined but reported to be in the vicinity of 90,000-100,000. Thus, a discrepancy exists between information provided by the genome and the proteome, a fact that has been observed experimentally for both eukaryotic and prokaryotic organisms. For instance, it is postulated that 40-60 percent of human genes have alternative splice forms (i.e., after transcription from DNA to ribonucleic acid (RNA), gene transcripts can be spliced in different ways prior to translation into proteins).

Genome sequences also do not elucidate post-translational modifications where many eukaryotic and some prokaryotic proteins undergo chemical modifications such as addition of carbohydrate and phosphate groups. Consequently, in the cases of alternate splicing and PTMs, the information from a single gene may encode many different proteins.

Rather than using genomics information, we propose to unravel proteomes by directly sequencing proteins. To this end, we have developed and benchmarked a novel technique based on multiple enzymatic digestion of a protein or protein mixture that reconstructs protein sequences from sequences of overlapping peptides. This approach, analogous to shotgun sequencing of DNA, is to be used to sequence alternative spliced proteins, to identify post-translational modifications, and to sequence genetically engineered proteins.

Our approach is composed of the following steps:

- Select and purify the proteins of interest.
- Cleave the proteins in serial and parallel reactions using enzymatic digestion.
- For products of each reaction, measure peptide masses and ion masses using tandem mass spectrometry (MS/MS).
- For each MS/MS spectrum, search peptides using several peptide identification codes.
- Compute a score matching the in silico and experimental MS/MS spectra.
- In case of poor score, subject the MS/MS spectrum to de novo sequencing, which is used to uncover full peptide sequences and partial sequence tags, which in turn are used to search in databases.
- The overlapping identified or de novo sequenced peptides are used by an assembly algorithm to de novo reconstruct protein sequences.

FY 2006 Accomplishments

We completed two milestones: 1) develop and benchmark the protocol for sequence reconstruction, and 2) perform an error analysis in order to develop a scoring function for peptide identification. Here are our results.

Develop and Benchmark Protein Sequencing Protocol
We selected carbonic anhydrase (CA-II) as the initial protein to be sequenced. Seven enzymes were identified as good candidates for multiple digestion based on their specific cleavage sites; those enzymes are trypsin, Lys-C, Glu-C, chymotrypsin, pepsin, proteinase K, and elastase.

After incubating CA-II with these enzymes in parallel, we acquired MS/MS data for the peptide products and submitted the data to the protein identification search engine MASCOT (MatrixScience, Boston, MA). We searched the spectra against the entire SwissProt protein to mimic the situation where the analyzed protein is unknown and then fed the identified sequences to our sequence assembly method.

We developed and coded an assembly algorithm based on finding an Euler path in a graph representing overlaps between the peptides and tested our assembly algorithm by performing an in silico digestion of *Escherichia coli* K12 proteins with an increasing number of enzymes. The assembly algorithm indicated that a large fraction, ≥75 percent, of protein sequences could be recovered when 10 or more enzymes are used in parallel. Our assembly algorithm was then used with the CA-II peptide identified by MASCOT. And, finally, we successfully assembled 82.6 percent of the sequence.

MS/MS Error Analysis of a Designed Peptide Array
We found that the algorithms used for peptide identification work better for tryptic-type peptides than for nontryptic ones. This is a problem because in this project we digest the protein of interest using multiple enzymes of different specificities. Therefore, to accomplish our goal we require an understanding of how peptides dissociate based on charge state, amino acid composition, length, and N- and C-terminal characteristic. To this end, we designed and synthesized an array of 40 peptides, each of length 11.

We designed the array such that each amino acid pair would be represented exactly once and subjected each peptide to MS/MS analysis where the product ions were assigned to their peptide bond cleavages. We grouped the intensities of each bond cleavage by bond type and determined the median intensity value of each bond type. We used the ratio of the median intensity of each bond type to the median intensity of all bond types to determine whether certain amino acids tended to enhance or suppress cleavage of adjacent bonds. Using this ratio, we reproduced the most commonly reported features in the literature of enhanced cleavage and suppressed cleavage. We found several yet unpublished patterns, such as the suppressed nN cleavage and the enhanced nH cleavages.

Significance

The primary method in proteomics analysis is protein identification by MS. A typical identification technique consists of enzymatic cleavage of one or more proteins followed by separation and MS or MS/MS measurements of the resulting peptides. All protein identification codes are plagued by false-positive peptide sequence identifications. To overcome the problem of protein identification, we developed *de novo* protein sequencing using MS/MS data but are still limited to short sequences.

We developed and benchmarked a novel method allowing *de novo* protein sequencing that appears to be as accurate as traditional sequencing methods, while being as fast as identification techniques. The idea driving our method is to use the shotgun sequencing method developed for genomic analysis but mostly unused in proteomics.

The impacts of accurate protein sequencing span several DOE, Department of Homeland Security, and Sandia interests, as the technique is crucial for detecting engineered toxins, characterizing proteins for biofuels and decontamination, and providing sequences for databases of biowarfare agents. Sequencing and identification of presently unknown toxins, and genetically engineered variants of known toxins, that can be potentially used in biological warfare is of great interest to national security. Sequencing of these toxins will not only allow for identification and detection but will also enable us to build a new generation of inhibitors and receptors for these toxins.

Refereed Communications

S.P. Gaucher, A.K. Singh, and J.M. Faulon, “Non-Tryptic Peptides,” presented at Asilomar Conference on Mass Spectrometry, Pacific Grove, CA, October 2006.

S.P. Gaucher, A.K. Singh, and J.M. Faulon, “Tandem Mass Spectrometry of Nontryptic Peptides,” presented at American Society for Mass Spectrometry, Seattle, WA, May 2006.

Critical Advances in Cognitive Science and Technology: The Cognitive Collective and Cognitive Science and Technology Foundations

101931

A. E. Speed, K. R. Dixon, S. J. Verzi, M. C. Chen, C. C. Dornburg, J. T. McClain, W. Shaneyfelt, P. G. Xavier, B. E. Hart

Project Purpose

The primary purpose of this project was to create an upgraded version of the Cognitive Collective, a set of algorithms that enables individual expert models to interact with one another in a human-like manner and allows us to simulate group decision-making among a group of experts. This capability was initially developed with LDRD funding and was included in the Next-Generation Intelligent Systems Grand Challenge (LDRD Project 41194, FY 2003-2005).

Despite early promising success with the Collective, we identified a number of technical issues that needed to be in place in order to make the Collective applicable in a more real-world setting. The purpose of this project was to add capabilities and integrated features to make the Collective more readily applicable to real-world problems.

FY 2006 Accomplishments

- Conducted experimental assessment of model frameworks
- Integrated weighted sum Collective algorithms with a hybrid Java/C++ distributed version of Collective extensible for use with multiple cognitive architectures
- Created high-level ad hoc Collectives design
- Conducted additional research on group decision making and computational modeling of group decision making

We conducted a comparison between Sandia's Cognitive Framework and the leading cognitive framework from academia, ACT-R. This comparison used results from work we performed with the Defense Advanced Research Project Agency (DARPA) augmented cognition program and consisted of hiring a small company (Adaptive Cognitive Systems, LLC) to build an analogous ACT-R model using the driving data collected for the DARPA project. The general results from this comparison indicated that two types of ACT-R framework (rule-based and instance-based) agree with human raters of driving data at approximately the same rate that the Sandia framework does. However, we found that the ACT-R framework is less able to handle large amounts of data in reasonable timeframes, thereby indicating that for real-world applications, the Sandia framework is more appropriate than the ACT-R framework.

The integrated version of the Collective we created in FY2006 includes a couple of important features.

- The Collective itself is a framework that can accept any type of cognitive architecture so long as that architecture conforms to the defined interfaces. To this end, we could theoretically have a Collective that comprises Sandia models, ACT-R models, SOAR models, or whatever other types of architectures are most appropriate.
- This version of the Collective is capable of being distributed across multiple machines and locations.

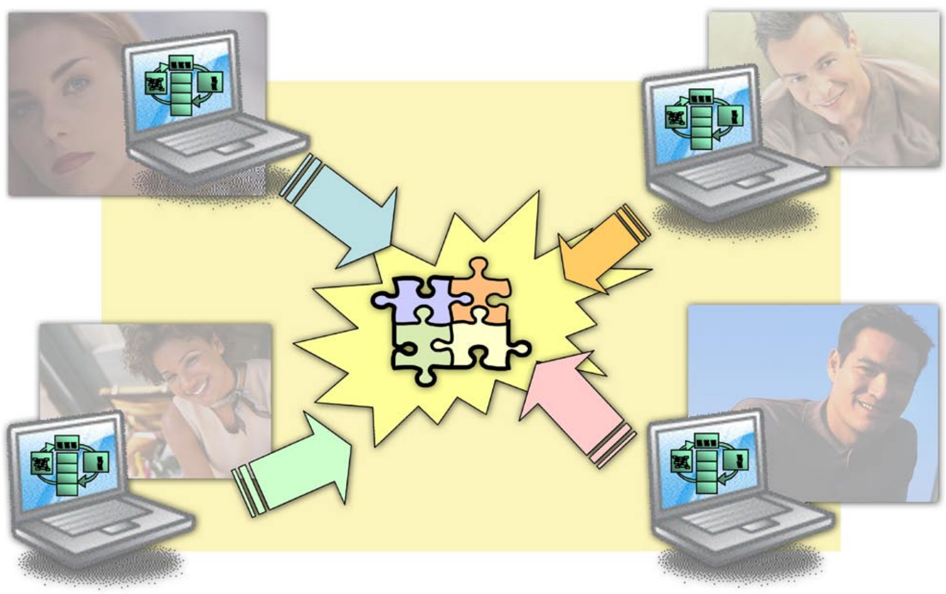
We added significant capability to the Collective and more completely integrated features of the prior three versions into a single piece of software. These upgrades along with the integration make the Collective more readily applicable to real-world problems than it had been in the past.

Significance

Based on our detailed research of the literature, the Cognitive Collective is one of only a small handful of current computational models of group decision making. However, the other existing models are academically oriented, and are therefore not intended for application to messy, large, real-world problems. To this end the Collective represents a significant, novel accomplishment.

While the Collective was initially built for dealing effectively with large data sets, as we have investigated a variety of data mining technologies, and as we have unearthed some of the technical issues still existing with the Collective, we believe that the most effective application for the current and near-future aspects of the Collective will be in problems for which information is distributed across different data sources (e.g., “hidden profiles”) or for which information is stovepiped (either intentionally or unintentionally). Because of the distributed nature of the Collective, different models can reside on different machines or in altogether different locales.

In this manner, as the Collective becomes more mature, it could begin to solve “hidden profile” types of problems where the relevant information for solving a problem or for developing an overall picture of ongoing events are distributed across time and data sources/data source locations. This could have significant benefit for intelligence and homeland security problems.



Distributed Cognitive Collective: Cognitive models of experts developing a more complete situation understanding despite being distributed across multiple machines

Nano-Bio-Cogno Convergence Concept Design Study

103396

J. C. Forsythe, H. Fan, K. R. Dixon

Project Purpose

Technologies in the nanotechnology, biotechnology, and cognition domains have advanced to the extent that their convergence opens opportunities for significant technical innovations. Sandia is uniquely poised with successful initiatives in each area. This project serves as a first step in bridging Sandia's initiatives in these domains.

FY 2006 Accomplishments

We conducted interviews to characterize Sandia's expertise in the nanotechnology and biotechnology domains. The cognition domain was not included in these interviews because a parallel study has characterized Sandia's expertise in this area and will document these findings in a separate report.

As follow-up to the interviews, we held a day-long workshop where experts from the nanotechnology, biotechnology, and cognition domains participated in brainstorming technical concepts, and a smaller group of workshop participants evaluated these concepts. One objective of the workshop was to develop partnerships with the University of New Mexico (UNM), the UNM MIND Institute, and the Beckman Institute at the University of Illinois. Each of these institutions was represented at the workshop and within the core group that evaluated the generated ideas.

Significance

Through the development of technical concepts for future research projects involving a convergence of Sandia strengths in the nanotechnology, biotechnology, and cognition domains, this project provided significant input into Sandia's cognitive systems and technology planning document and, in particular, technical roadmap.

Cognitive Science and Technology Synergies Concept Design Study

103925

E. P. Parker, W. Shaneyfelt, W. A. Stubblefield

Project Purpose

A young discipline, cognitive science is characterized by different theoretical schools and methodologies. The only thing these schools have in common is a commitment to computational representation of cognitive processes, a fact that hinders both communication and research.

The goal of this project was to establish a common foundation for Sandia's cognitive science community to build capabilities, to enhance collaboration and communication among different researchers, and to provide a common theoretical baseline for evaluating work. During the course of this project, we investigated the full breadth of Sandia's technical contributions to the cognitive science and technology (CS&T) area. Identifying areas where projects converge and novel opportunities exist is difficult given the diverse cognitive science activities at Sandia. We addressed this problem by bridging Sandia initiatives in cognitive science.

FY 2006 Accomplishments

- We conducted a workshop titled "Cognitive Science & Technology Synergies: Brainstorming Technology Futures."
- We compiled a survey of cognitive science-related projects and expertise at Sandia.
- We produced a set of potential new directions and recommended actions for building CS&T capabilities.
- We documented our results in a SAND report.

Significance

The results of this project were used in Sandia's CS&T strategic planning document, which defines actions that need to be followed in order to pursue internal collaborations between program areas and external collaborations with universities and research institutes.

Our survey of existing projects that relate to cognitive science at Sandia provides decision makers with a clearer picture of the wide-ranging potential CS&T has to affect Sandia's future missions.

We identified actions that management needs to take to support the development of CS&T capabilities at Sandia. If met, these actions will increase Sandia's ability to 1) publish in respected academic journals, 2) collaborate with the academic community, 3) affect the direction of research and development in the academic community, and 4) support future national security missions.

Neural Interface Evaluation

104735

B. R. Rohrer

Project Purpose

The purpose of this project was to develop novel neural interface concepts. We worked closely with the University of New Mexico (UNM) MIND Institute and the UNM BRaIN Center to build our partnering capabilities and to leverage their neurophysiology and neuroscience expertise. We planned to develop two concepts: one that might be potentially feasible using current technology, and one that may be feasible given a concerted five-year development effort.

FY 2006 Accomplishments

Surveyed existing neuroimaging techniques

Scientists at the MIND Institute and the BRaIN Center provided Sandia researchers with a technical overview of neuroimaging techniques currently in use or under development. Follow-up discussion focused on the possibility of miniaturizing and making portable the required apparatuses.

Brainstormed neural interface concepts

We generated a list of possible neural interface concepts, emphasizing concepts not currently in use or dramatically different from anything that has been so far proposed.

Selected and refined concepts

We selected the most likely of the brainstormed ideas and determined specifically what technological developments would be required before each one became feasible. We constructed a rough timeline for each idea.

Surveyed existing cognitive models

In order to interact with the central nervous system, an understanding of how it functions and of how those functions are related to its anatomy was required. We performed a survey of existing cognitive models in order to determine to what extent such a mapping between function and anatomy has been performed.

Significance

The significance of neural interface development lies in the ability of humans and machines to interact seamlessly. If successful, a high-performance neural interface would allow machine hardware to assist a human in a very organic way.

Studies in psychology indicate that when a machine moves in response to mental commands, and when interaction of that machine with the environment produces a natural sensory experience in the user, the user begins to incorporate that machine into his/her mental model of his/her body. In this way advanced neural interfaces may allow the "extension" of users, whether to compensate for injury or age, to assist with demanding tasks, or to extend their power and capability.

Foundations for Augmented Cognition Systems that Use Nanoscale Materials

104737

H. Fan, E. P. Parker

Project Purpose

The purpose of this "concept definition" project is to support future research using nanomaterials as a basis for observing and measuring neurophysiological processes. Through this work we hope to gain scientific insights concerning network properties associated with neural representations and processes and establish the foundation for expanding current capabilities to create nanoelectrode arrays that will allow nanoscale measurement of the activity of tens to hundreds of neurons.

FY 2006 Accomplishments

We completed a report summarizing our background research that will provide a foundation for work planned for FY 2007 and beyond. Two important studies in our report are: 1) the neuroanatomy and neurophysiology of the hippocampus, and 2) programs of research that have addressed the relationship between neurophysiological processes and behavioral measures of memory performance. These studies provide insight into methodological and analytic approaches for studying the representation of memory processes in the hippocampus.

Significance

This project accomplished key foundational work in preparation for future projects aimed at applying Sandia nanoscale capabilities to neurotechnology.

The Effects of Angry and Fearful Emotion States on Decision Making

104742
M. L. Bernard

Project Purpose

The purpose of this study was to better understand the role of emotion states and traits in making decisions that involve varying degrees of risk. Emotions have evolved as action tendencies that enable humans to react quickly and effectively to prototypic situations. Successful decision making is critical as humans adapt to their environment. It can involve choosing between competing actions associated with different magnitudes of reward/punishment or different levels of probability of benefits/penalties.

Anger and fear, as part of the fight or flight response, may have an especially strong influence on the decisions people make in risky situations. These emotions are involved in responding in emergency situations. Understanding the role of these emotional states is critical for predicting what decisions people will make in situations such as terrorist attacks, natural disasters, pandemics, and combat. Unfortunately, there is a current lack of understanding of how emotional states influence decision making, particularly making safe versus risky choices.

To study how emotion traits and states influence decisions, researchers from the University of New Mexico (UNM), the UNM MIND Institute, and Sandia used behavioral and neuroimaging methods to examine the behavioral and neural responses of 26 healthy individuals (13 men and 13 women) as they underwent functional magnetic resonance imaging (fMRI) while engaged in decision-making tasks.

The two primary tasks given them were: trying to win money by choosing between alternatives that offered less money for less risk and more money for more risk, and trying to avoid losing money by choosing between alternatives that involved a greater risk of losing a small amount of money and a lesser risk of losing a larger amount of money.

We measured trait anger and trait anxiety (as a measure of generalized fear) to determine how they were related to making safe versus risky choices. We also measured and examined the role of state anxiety and state positive affect as emotion states that may influence decision making.

Making safe versus risky choices in relation to winning money

We hypothesized that trait anger would be related to taking more risks, and that trait and state anxiety would be related to taking fewer risks. In addition, we predicted that positive affect would be related to less risk taking.

Making safe versus risky choices in relation to losing money

We hypothesized that trait and state anxiety would make people less willing to risk losing more. We had no hypotheses regarding trait anger and positive affect.

Neural results

We predicted that safe choices would be associated with activity in brain regions associated with positive emotions, including the left prefrontal cortex and nucleus accumbens. Finally, we predicted that risky choices would be associated with activity in brain regions associated with negative emotions, including the right prefrontal cortex and the amygdala.

FY 2006 Accomplishments

We used fMRI to examine the influence of emotion traits and states on decision making. Half of the experiment was called the “winning game” in which participants could either win or not win money. During this game, participants were confronted with having to choose between a small chance of winning a lot of money (risky choice) and a large chance of winning a little money (safe choice).

We found that trait anger was strongly related to making riskier decisions ($r = 0.52$, $p < 0.01$), and that both trait and state anxiety were related to making riskier decisions ($r = 0.39$, $p < 0.05$ and $r = 0.42$, $p < 0.05$, respectively). In addition, state positive affect was related to a tendency to make less risky decisions ($r = -0.46$, $p < 0.05$). During the trials in which the risk and reward were nearly balanced, the correlation between trait anger, trait anxiety, state anxiety, and positive affect were nearly the same (0.58, 0.47, 0.51, -0.43, respectively). These results suggest that emotional states and traits may have a strong influence on decision making involving risk.

The other half of the experiment was called the "losing game" in which the participants could either lose or not lose money. During this game, participants were confronted with having to choose between a small chance of losing a lot of money and a large chance of losing a little money. We found that trait anger and state anxiety were both related to a strong tendency to select the large chance of losing little money ($r = 0.50$, $p < 0.01$ and $r = 0.46$, $p < 0.05$, respectively).

Several other noteworthy results were not related to the hypotheses. First, we found that greater age was related to the tendency to make safer choices when the stakes of the decision were higher ($r = 0.52$, $p < 0.01$). Second, we found a general bias toward making safer choices, even when riskier choices would produce better results. Third, we found that reaction times were slower for women than men during trials in which the choice was not as easy (when risk was nearly equally balanced with reward) and for trials in which the stakes were higher.

Overall, the scientific impact of this study was that it identified that potentially dangerous biases could be caused by emotion traits and states in decision making. This knowledge may also make it possible to identify individuals who are influenced by anger and fear in harmful ways. Further, this study will serve as a model for examining the influence of emotion, personality, and culture on decision making.

Significance

The significance of the behavioral results is that anger, anxiety, and positive affect may all have important implications for decision making. In short, people higher in anger (trait) and anxiety (trait and state) were more likely to make risky decisions, and people higher in positive affect were more likely to make safe decisions.

These results have strong implications for people making decisions in emergencies and critical situations such as first response to disasters, terrorist attacks, and epidemics and wartime situations such as combat. That is, those who are higher in anger and anxiety may be more likely to engage in risky behaviors. The fight or flight response may be more mobilized during these emotions, making it more likely that people take risks.

In contrast, positive affect may serve to reduce the likelihood of engaging in risky behavior, perhaps because the person is already feeling good and feels less of a need to risk losing that state. With regard to an overall model of the effects of emotion on risky versus safe decision making, it may be that negative emotions contribute to the ability to make risky decisions when necessary, and positive emotions contribute to the ability to make safe decisions when necessary.

Future directions that may be taken in continuing this line of research include:

- Confirming these results in situations that more closely resemble the kinds of real-life situations of interest to Sandia.
- Closely examining the anger in its trait and state forms since it seems to be so important and since we only examined trait anger in this study.
- Examining interventions that might enable people to better use their emotions in decision making in critical situations. If both positive and negative emotions can play an important role of decision making, then it may be important to find ways to enable people to be more aware of their emotions and to use them optimally when making an important decision.

Mindfulness techniques may be used to increase awareness of positive and negative emotions, to distinguish between those that are relevant to the current decision versus distractions, to weigh the range of positive and negative emotions in making the best decision, and to focus on the emotions that will most likely enable the person to carry out the choice.

We await the neural results to better understand how the brain supports risky versus safe decisions and to identify the brain circuits through which emotion influences these choices. At that time, we will determine whether there are differences in neural activity between those who are high, versus low, on positive and negative emotions. And we will specifically focus on regions that support these emotions, effective decision making, and emotion awareness and regulation.

fMRI Analysis of the Decision-Making Processes of Human Subjects

105305
J. T. McClain, S. J. Verzi

Project Purpose

A key objective of the cognitive science and technology (CS&T) focus area at Sandia is to develop a better understanding of the psychological and biological processes that support human cognition. An important part of this effort is to understand the basic functions involved with human learning. An improved understanding of human learning will not only advance the science of human cognition but could also improve our ability to computationally model human cognition, which would have wide-ranging benefits.

Function learning involves gaining knowledge about an association between two sets of numbers. Learning the conversion between Fahrenheit and Celsius temperature scales is one case of function learning. While there has been a fairly broad effort toward developing function learning in machines, both in the area of machine learning and in the area of function approximation, much of this work is focused on improving function learning without regard to the biological or psychological plausibility of such algorithms. A better understanding of the underlying processes associated with human function learning would help improve our ability to model human learning in general.

This project builds on some carefully developed (and traditional) psychological studies by McDaniel and Busemeyer [1, 2]. These tests divided human subjects into two major groups when they were asked to solve a simple puzzle with parameters that were represented by a mathematical relationship. During the training phase, subjects were given an input value and asked to guess the correct output value. The relationship between the input and output values was determined by a simple mathematical relationship, which the subjects did not know. They were then given feedback about the accuracy of their guess and, over a series of trials, were able to learn the relationship. During the test phase after training, they were given input numbers outside the range of their training and asked to guess the correct output value.

One group of subjects, the exemplar learners, simply (and repeatedly) responded with the output value for the closest input value they had learned. The second group, the extrapolator learners, was able to discern and apply the underlying mathematical relationship involved in their training session and extrapolate it to any new numbers, even those outside their original range of training.

The purpose of this project was to extend the work of McDaniel et al. in order to develop a better understanding of the brain functions involved with human function learning and to explore possibilities for the computational modeling of human learning.
We proposed three primary objectives:

- Attempt to identify the neural correlates of the human learning subgroups through an in-depth analysis of the human fMRI data.
- Attempt to develop computational models that characterize the differences between the two learning subgroups.
- Use the knowledge gained from objectives 1 and 2 to outline future experimental efforts for examining human function learning.

FY 2006 Accomplishments

- We identified the cortical components correlated with the human learning subgroups and

[1] M.A. McDaniel and J.R. Busemeyer, "The Conceptual Basis of Function Learning and Extrapolation: Comparison of Rule-Based and Associative-Based Models," *Psychonomic Bulletin & Review,* vol. 12 (1), pp. 24-42, February 2005.
[2] J. Busemeyer, M.A. McDaniel, and E. Byun, "The Abstraction of Intervening Concepts from Experience with Multiple Input-Multiple Output Causal Environments," *Cognitive Psychology,* vol. 32**,** pp. 1-48, February 1997.

discovered time differentials between learning subgroups when generating responses.

- We began an exploratory analysis of a variety of machine learning algorithms, with comparisons to the human learning subgroups. We also developed a classifier that was capable of differentiating the two learning subgroups based on the behavioral data collected in the experiment.
- We identified a number of experimental pathways to extend our current understanding of human function learning, including a need for additional temporal analysis of the cortical components involved in function learning using EEG, the exploration of the emotional aspects of human function learning (i.e., the limbic system), and a further analysis of crossover subjects.

The first and most important result of this effort was establishing strong correlations between the strategies of groups of rote and function learning human subjects and critical areas of cortex. McDaniel et al. established that groups of human subjects adopted different strategies when trying to learn mathematical relationships. One group of subjects took a more "rote learning" or "memory-based" approach to the problem, while the second group showed a marked effort toward the "functional (mathematical) relationship." We refer to these groups as taking the "rote" or "functional" approaches to learning the mathematical relationship.

We made several important discoveries:

- We identified four subjects who changed their solution strategies during the problem-solving process. Seeing these subjects and analyzing their changes in solution strategy, as well as the corresponding changes in cortical patterns, opens up a number of interesting questions for continued research.
- We detected changes in the subjects' response times during the trials. The subjects with the rote learning responses required fairly constant time periods for generating their solutions. The function learning subjects, however, required decreased response times across successive trials. These differences also were reflected in their corresponding cortical activation. These time differentials, we believe, reflected the subjects' improved skills at generating appropriate functional relationships between the experiment's cues and the subjects' responses.
- We observed a temporal difference in the convergence of subject accuracy during the training phase. This allowed the development of a classification algorithm that classified the different learning groups with an accuracy of 80 percent using only the subjects' training data. The ability to separate these groups with such a small number of subjects is a very significant achievement.

Significance

This project has contributed to the general knowledge of human function and rote learning behavior. In particular, we identified the key cortical components associated with two different types of function learners: exemplar learners and extrapolator learners. This project also led to the discovery of human behavioral measures that can be used to distinguish these types of function learners with high accuracy.

An enhanced understanding of the cortical areas associated with human function learning will have a significant impact on one of Sandia's focus areas: human . With continued research into this area, Sandia will be able to develop a better understanding of the functional processes associated with learning, which could lead to better modeling of human learning on computers.

The ability to separate human learners based on the learning strategy they are using during the training phase could have huge impacts on future training techniques. For example, a future training system could identify when a person was using a less-than-ideal strategy for learning as the training was taking place and possibly redirect them toward a more viable strategy.

Greater knowledge of human learning processes and behaviors could help improve human error detection and augmentation technology, allowing for better detection of human error in the areas of physical security and nuclear weapons safeguards, along with other mission critical applications.

Investigating Surety Methodologies for Cognitive Systems

105306

D. E. Peercy, W. Shaneyfelt

Project Purpose

The purpose of this project was to identify and characterize the risks associated with cognitive science technologies, as well as examine appropriate surety levels and relevant approaches that can be used to mitigate these risks.

Recently, numerous highly divisive scientific controversies have constrained, even halted, technology advancements (e.g., the Waste Isolation Pilot Plant, Yucca Mountain repository, stem cell research). While some controversy is based on technical risk, other hindrances are due to perceived risks such as public reaction to biased media reports.

This feasibility study was conducted by Sandia and University of New Mexico (UNM) personnel with the expertise to examine the emerging science of cognition and characterize a range of actual, potential, and perceived technical and nontechnical risks. The study consisted of three phases.

Phase 1: Conduct interviews with small focus groups to identify the spectrum of risks for identified cognitive systems. We identified five focus groups: baseline, nontechnical, technical, public, and surety; surveys and discussion sessions elicited a spectrum of risks related to the development and implementation of cognitive systems.

Phase 2: Identify techniques and tools. The surety focus group, consisting of Sandia surety engineers, completed the survey tool and discussion and addressed surety levels and methodologies that might apply to cognitive systems based on the spectrum of risks identified in Phase 1. Of particular interest were techniques to use as integrated mechanisms within the design of cognitive systems.

Phase 3: Publish the results of Phase 1 and 2 in a SAND report that can serve as a model for others in the global technical community, as well as provide input for new national initiatives. Recommendations are provided for further study based on the results of this short LDRD effort.

FY 2006 Accomplishments

Phase 1: Study members Eva Caldera (UNM) and Wendy Shaneyfelt (Sandia) gave a poster presentation, “Where Do You Draw the Line?” at a cognitive systems workshop in Santa Fe, NM. A survey tool was developed for distribution at this presentation, and initial results established a baseline that could be used to compare with selected focus group results. This work and comments from participants fed into a subgroup led by Wendy Shaneyfelt at a cognitive dominance workshop in Washington, DC.

A focus group procedure was developed and applied to the subsequent focus groups meetings: 1) nontechnical (UNM professors) focus group; 2) public focus group; 3) technical (cognitive scientists) focus group; and 4) Surety focus group. Each group completed the survey questionnaire and participated in a recorded discussion on the topic of risk related to cognitive systems. Survey responses were entered into an Excel spreadsheet, the discussion recordings edited for anonymity, and a summary of the main discussion points created.

Phase 2: The surety focus group reviewed a spectrum of risks identified by the previous focus groups. Surety method and technologies for safety, security, reliability, support/sustainment, and quality were provided and linked to potential use in reducing the level of risk in the various identified spectrum categories.

Phase 3: The results of this study, along with recommendations for continued research in this area, are being documented in a final report.

Significance

Although this study was short, several key results regarding the risk spectrum and potential application of surety methods could have significant impact to the general scientific community and to Sandia's mission areas. Cognitive systems can have a significant role in areas such as prognostics, health management, neurosystems (understand, restore, enhance, degrade), and microsystems (mechanical, biological, chemical). Research is being conducted in and products produced for military, commercial, health, legal, sociological, medicine, education, economics, and human enhancement.

Understanding the potential risks (perceived and actual) of the cognitive system technology in those applications is key to effective application. The surety methods that have been successfully applied to weapon and weapon-related products, as well as research efforts in related areas such as uncertainty quantification and risk-based decision processes, have significant potential for assisting decision makers in quantifying and reducing the risk of failure and misuse.

Further study is needed to construct a framework within which cognitive systems can be identified, studied, analyzed for potential risks, and designed for acceptable application risk. This initial study provides a basis for conducting further research and links the surety methods to potential risk-reduction application.

Biological Research Survey for the Efficient Conversion of Biomass to Biofuels

105674
M. S. Kent

Project Purpose

The purpose of this “concept definition” project was to investigate the science and technology of economical biofuel production. The main focus was on producing bioethanol from lignocellulosic biomass and biodiesel from nonpetroleum sources, mainly vegetable oils and algae. We had three main goals: 1) assess the major technological hurdles for economic production of biofuels for these two approaches, 2) compare the challenges and potential benefits of the two approaches, and 3) determine areas of research where Sandia’s unique technical capabilities can have a strong impact in both of these technologies.

FY 2006 Accomplishments

- Completed in-depth review of recent reports on biofuels:
  - o DOE Office of Biological and Environmental Research/Genomes to Life Biomass to Biofuels Workshop report (June 2006)
  - o National Renewable Energy Laboratory (NREL) report - Biomass Oil Analysis: Research Needs and Recommendations (June 2004)
  - o NREL closeout report - A Look Back at the U.S. Department of Energy’s Aquatic Species Program: Biodiesel from Algae (July 1998)
- Completed comprehensive review of scientific literature and patent search
- Engaged Sandia researchers in discussions regarding application of technical expertise to specific technological problems related to biofuels production

Significance

This research will help Sandia management to map Sandia’s technical strengths onto technology areas of high impact for biofuel production.

Chemical & Earth Sciences

Poroelastic Wave Propagation Modeling and Inversion

69198

D. F. Aldridge, C. J. Weiss, L. C. Bartel, U. Hetmaniuk, N. P. Symons, B. G. van Bloemen Waanders

Project Purpose

The primary purpose of this project during FY 2006 was to improve and refine previously developed numerical algorithms for simulating three-dimensional (3D) seismic wave propagation through a poroelastic (fluid-saturated solid) medium.

Using fundamental principles of Biot theory, we developed a system of 13 coupled first-order partial differential equations governing wave propagation within a porous medium. Numerical solution methodology consists of explicit, time-domain, finite-differencing of these expressions on staggered spatial and temporal grids. Finite-difference operator accuracy is second-order in time and fourth-order in space and thus provides a reasonable compromise between computational complexity and numerical accuracy of the computed results.

A single-processor workstation version of the algorithm is suitable for development, testing, and validation purposes. Moreover, small-to-intermediate poroelastic modeling scenarios can be conducted in reasonable execution times with this serial version of the algorithm. However, large-scale modeling (i.e., hundreds of millions of spatial gridpoints and tens of thousands of timesteps) demands a massively parallel numerical algorithm. Much of our effort during FY 2006 was devoted to implementing various algorithmic improvements in the parallel algorithm.

Although the principal objective of this project was to develop poroelastic wave propagation modeling tools, we also initiated several application modeling examples designed to validate the algorithm and enhance our understanding of poroelastic effects in recorded seismic data. Numerous tests verify that the algorithm generates correct results in the two limiting (and problematical) cases of vanishing porosity (i.e., an elastic medium) and unity porosity (i.e., an acoustic medium). Finally, extensive numerical simulations of poroelastic waves recorded by shallow subsurface crosswell acquisition geometry allowed us to understand the characteristic features of poroelastic wave arrivals in seismic data.

Interestingly, the slow compressional (P) wave, mathematically predicted by Biot theory, is calculated with fidelity by our algorithm when the viscosity of the pore-saturating fluid (brine) is set equal to zero. However, a realistic viscosity value for water (0.001 Pa_s) attenuates the slow P wave so much that it cannot be observed in the calculated data. This is probably why the slow P wave has rarely (if ever) been observed in field-recorded seismic data. Nevertheless, successful observation and use of the slow P poroelastic wave may yield significant enhancements in the resolving power of seismic exploration methods.

FY 2006 Accomplishments

Developed a superior poroelastic model construction algorithm

The partial differential equations (PDEs) governing linearized wave propagation through a two-phase (fluid-saturated solid) medium depend on nine material parameters. These parameters appear as coefficients in the fundamental PDEs. However, parameters are complicated nonlinear functions of quantities that are more familiar to an exploration seismologist.

We implemented a model construction algorithm where a user specifies well-understood quantities (composite medium compressional and shear wavespeeds, mass density) that are subsequently reformulated into parameters required by the poroelastic computation algorithm. Moreover, this model construction approach correctly handles the two problematic cases of zero porosity (elastic medium) and unity porosity (acoustic medium) without recourse to time-consuming conditional logic in the subsequent response computation algorithm.

Incorporated additional seismological functionality into the serial version of the finite-difference poroelastic wave propagation algorithm
Body sources of seismic waves are considered either force densities or moment densities. Sources are applied to either the fluid, solid, or both constituents of the poroelastic medium. Similarly, seismic energy receivers are receptive to motion (particle displacement, velocity, acceleration, pressure, rotation) of the solid, fluid, or composite phases. Timeslice displays, extremely useful for interpretation of the seismic wavefield, are also implemented. It is emphasized that receivers/timeslices recording particle rotation, designed to isolate shear wave arrivals of the total seismic wavefield, are an extremely novel aspect of numerical wave propagation algorithms.

Implemented the aforementioned improvements to the finite-difference (FD) algorithm within the massively parallel version of the algorithm
We are implementing the enhancements to the serial version of the poroelastic wave propagation algorithm in the analogous parallel version. Treatment of large-scale poroelastic wave propagation scenarios requires such parallel computation capability.

Implemented a stress-free/pressure-free boundary condition, mimicking conditions at the earth's surface
The most important reflecting interface in all seismic wave propagation problems is the earth's surface. Introduction of this particular boundary condition into our simulation algorithms enhances seismological utility.

Conducted verification tests to ensure compatibility of results calculated by the poroelastic algorithm in the two important limiting cases of vanishing porosity and unity porosity
Our poroelastic algorithms should generate results identical to existing FD codes for elastic and acoustic waves. After much detailed algorithm validation and vetting work, we find that this is indeed the case.

Developed a poroelastic Green function algorithm, applicable to a homogeneous and isotropic wholespace
This algorithm provides an essential check on the veracity of results calculated by the time-domain FD approach. Additionally, rapid forward modeling of poroelastic wave propagation effects is readily accommodated (i.e., within seconds, rather than hours, of execution time).

Modeled CO_2 sequestration scenarios
Sequestration of CO_2 in underground geologic repositories is an important element in evolving strategies for accommodating atmospheric CO_2 accumulations. Seismic monitoring of the sequestered CO_2 (in amounts of millions of tons) is considered the most favorable approach. Poroelastic wave propagation theory and numerical algorithms are well-suited for such seismic modeling investigations.

Significance

Accurate poroelastic wave propagation modeling is of great interest to the US petroleum industry for oil and natural gas exploration, production, and/or reservoir monitoring purposes. Hence, this project addresses a critical national security issue associated with domestic fossil energy resources.

Numerous applications involving remote detection and characterization of subsurface fluids and flow will also benefit from poroelastic simulation tools and modeling expertise. These include sequestration of carbon dioxide within depleted petroleum reservoirs or saline aquifers, migration of environmental contaminants within the shallow subsurface vadose or saturated zones, exploration for and salinity assessment of deep hydrological resources, and earthquake hazard prediction associated with fluid-controlled fault rupture.

Defense-related applications include assessment of penetration of an air-deployed weapon into shallow subsurface geologic strata, since penetrability is strongly influenced by formation porosity and saturation. Finally, the problem of large-scale poroelastic wave propagation simulation is ideal for development, testing, and validation of new massively parallel computational algorithms.

Refereed Communications

K.T. Kim, D.F. Aldridge, and N.P. Symons, "Seismic Wave Propagation in 3D Randomly Heterogeneous Elastic Media," presented at the Society of Exploration Geophysicists, Houston, TX, October 2006.

M.M. Haney, J.T. Fredrich, B.J. Zadler, J.A. Scales, and D.F. Aldridge, "Numerical Simulation of Resonances in Microtomographic Models," presented at the Society of Exploration Geophysicists, New Orleans, LA, October 2006.

N.P. Symons, D.F. Aldridge, and M.M. Haney, "3D Acoustic and Elastic Modeling with Marmousi2," presented at the Society of Exploration Geophysicists, New Orleans, LA, October 2006.

Other Communications

D.F. Aldridge, N.P. Symons, and M.M. Haney, "Finite-Difference Numerical Simulations in Seismic Gradiometry," presented at the American Geophysical Union, San Francisco, CA, December 2006.

L.C. Bartel, M.M. Haney, D.F. Aldridge, N.P. Symons, and G.J. Elbring, "Modeling of Time-Lapse Seismic Reflection Data from CO_2 Sequestration at West Pearl Queen Field," presented at the American Geophysical Union, San Francisco, CA, December 2006.

M.M. Haney, D.F. Aldridge, and N.P. Symons, "Numerical Dispersion, Stability, and Phase-Speed for 3D Time-Domain Finite-Difference Seismic Wave Propagation Algorithms," presented at the American Geophysical Union, San Francisco, CA, December 2006.

Developing the Foundation for Polyoxoniobate Chemistry: Highly Tunable and Exploitable Materials

70799

M. D. Nyman, R. P. Bontchev, T. M. Anderson, M. A. Rodriguez, T. M. Alam

Project Purpose

The purpose of this project is to develop the foundation of a new area of inorganic chemistry, polyoxoniobate chemistry. The discovery of new science that is documented in key publications is the product of this work. Polyoxoniobates are a subset of polyoxometalates (POMs). POMs are discrete metal-oxo inorganic clusters with intriguing characteristics that include high surface charge, super-acidic to super-basic behavior, redox chemistry, absolute discrete size and geometry that can be diversified by controlled solution chemistry, and ease of manipulation from solution to surfaces to solids. These properties are exploitable in catalysis, colorometric detection, corrosion inhibition, metal sequestration, pathogen precipitation, molecular magnets, luminescent sensing, and as building blocks for assembly of complex materials.

While the POMs of vanadates, tungstates, and molybdates have been well developed, the niobates are poorly understood and underdeveloped due to their more challenging synthetic chemistry and ability to be handled. Yet they warrant focused attention due to their unique characteristics as compared to their POM cousins. For example, POMs of V, W, and Mo are acidic, with a relatively low surface charge, while the polyoxoniobates are basic, and possess high surface charges. This makes the polyoxoniobates potentially optimal for any application involving electrostatic interactions or binding. We were the first to show that polyoxoniobates can have similar structural attributes as their POM cousins [1]. In this project, we seek to broaden the base understanding of these materials, in particular their structures and behavior.

FY 2006 Accomplishments

We synthesized polyoxoniobates with transition-metal linkers or transition-metal counterions instead of alkali metals. This is a synthetic challenge because the solution chemistry of transition metals (insoluble in base) is incompatible with the solution chemistry of polyoxoniobates (basic). It was achieved with Mn^{4+}, and Co^{2+} and Cu^{2+} by using coordinating ligands that aid in retaining these metals in solution in alkaline conditions (ethylenediaminetetraacetic acid (EDTA) and amines, respectively).

The series of Mn^{4+}-polyoxoniobate complexes with different alkali counterions demonstrate that the high charge of polyoxoniobates can be exploited to stabilize high oxidation states of transition metals; and the charge on Mn can vary from 3+ to 4+ as a function of the degree of ion pairing of the coordinating cluster with the counterion: less ion pairing results in stabilization of higher oxidation-state Mn. The Cu^{2+} serves as a cluster linker to form a series of magnetic polyoxoniobate materials with different degrees of Cu-Cu ferromagnetic coupling as a function of Cu-Cu distance.

The Cu^{2+} counterion also produced unprecedented cluster geometry; the largest polyoxoniobate cluster reported to date. This large cluster, along with a few examples from literature, illustrates that nonalkali counterions are the key to obtaining unique polyoxoniobate cluster types. However, removing alkali-counterions from polyoxoniobates proves a sufficient challenge due to extensive cluster-alkali ion pairing.

[1] M. Nyman, F. Bonhomme, T.M. Alam, M.A. Rodriguez, B.R. Cherry, J.L. Krumhansl, T.M. Nenoff, and A.M. Sattler, “A General Synthetic Procedure for Heteropolyniobates,” *Science*, vol 297(5583), pp. 996-998, August 2002.

We produced the world's first experimental evidence for metal-oxo cluster-alkali ion pairing in solution. The $[Nb_6O_{19}]^{8-}$ Lindqvist ion is not only the workhorse of polyoxoniobate chemistry; it also possesses the largest surface charge of all polyoxometalates. It exhibits extensive ion pairing in the solid state that varies as a function of alkali radius (Cs>Rb>K>Na>Li); there are hints that this pairing is also significant in solution.

We therefore carried out strategic experiments to directly observe ion pairing in solution. In particular, ^{133}Cs nuclear magnetic resonance (NMR) was used to investigate lifetimes of interaction between Cs and the $[Nb_6O_{19}]^{8-}$ Lindqvist ion. We also collected SAXS (small-angle x-ray scattering) data in collaboration with Mark Antonio (Argonne National Laboratory) to determine the radius of gyration in solution. This data is currently under careful evaluation.

Finally, we began to extend our knowledge of polyoxoniobate chemistry to a more challenging problem: polyoxotantalate chemistry. Polyoxotantalates are even less developed than polyoxoniobates. Currently, there is not even an aqueous, low-temperature synthesis to the only known cluster type, the Lindqvist ion $[Ta_6O_{19}]^{8-}$. We developed a reliable aqueous synthesis for the Lindqvist ion and are now attempting to expand this synthesis to produce heterometallic POMs that contain Ta^{5+}. In collaboration with Bill Casey (University of California at Davis) we are investigating the exchange rates of oxygen in the cluster.

These studies will provide insight into why polyoxotantalate chemistry is so different from polyoxoniobate chemistry, when it had been predicted to be so similar. When we complete and publish this work, it will be the first major advance in polyoxotantalate chemistry in about fifty years.

Significance

This work unquestionably contributes to Sandia's mission to carry out leading-edge science. Much of what we have discovered and reported on this project represents "firsts" of a very high order. We are the recognized world leaders in polyoxoniobate chemistry. New editions of textbooks and review papers on polyoxometalate chemistry will require significant updating due to our contributions to the field. Given the many exploitable properties of POMs, our work in this field has the potential to impact many areas of interest to Sandia and DOE.

Stabilization of high oxidation-state metals could be useful for catalysts that may contribute to the energy surety mission. The antiviral properties of POMs, as well as their ability to bind with charged biomolecules, may be exploited in the biosciences and homeland security. We are investigating coagulants for water treatment using POMs that are useful for removal of pathogens from potable or wastewater, a course of action that links to Sandia's water programs.

The super-basic characteristic of polyoxoniobates may be exploited for base-catalyzed decomposition of biowarfare agents, which links to homeland security and environmental remediation. Metal binding properties of polyoxoniobates are exploitable for nuclear waste remediation, as well as water treatment; which also links to the environmental missions of DOE and Sandia. The use of polyoxoniobates as nanometer-sized building blocks for assembly of complex materials is relevant to the Center for Integrated Nanotechnologies activities.

Refereed Communications

M. Nyman, T.M. Alam, F. Bonhomme, M.A. Rodriguez, C.S. Frazer, and M.E. Welk, "Solid-State Structures and Solution Behavior of Alkali Salts of the $[Nb_6O_{19}]^{8-}$ Lindqvist Ion," *J. Cluster Science*, vol. 17, pp. 197-219, June 2006.

M. Nyman, T.M. Alam, D. Ingersoll, H. Park, and J.B. Parise, "Polyoxometalate-Surfactant Arrays: The Magic #4," presented at Pacifichem 2005, Waikiki, HI, December 2005.

M. Nyman, "Polyoxoniobate Chemistry," presented at Pacifichem 2005, Waikiki, HI, December 2005.

J. Black, M. Nyman, and W.H. Casey, "Rates of Oxygen Exchange Between the $H_x[Nb_6O_{19}]^{8-x}$ Lindqvist Ion and Aqueous Solutions," to be published in *Journal of the American Chemical Society*.

F. Bonhomme, T.M. Alam, A.J. Celestian, D.R. Tallant, T.J. Boyle, B.R. Cherry, R.G. Tissot, M.A. Rodriguez, J.B. Parise, and M. Nyman, "Tribasic Lead Maleate and Lead Maleate: Synthesis, Structural, and Spectroscopic Characterizations," *Inorganic Chemistry*, vol. 44, pp. 7394-7402, October 2005.

R. Bontchev and M. Nyman, "Evolution of Polyoxoniobate Cluster Anions," *Angew Chem Int. Ed*, vol. Web release, pp. 1-4, September 2006.

M. Nyman, A.J. Celestian, J.B. Parise, G.P. Holland, and T.M. Alam, "Solid-State Structural Characterization of a Rigid Framework of Lacunary Heteropolyniobates," *Inorganic Chemistry*, vol. 45, pp. 1043-52, 2006.

Reverse-Time Seismic and Acoustic Wave Propagation: High-Fidelity Subsurface Imaging and Location of Energy Sources

79750

L. C. Bartel, S. J. Younghouse, N. P. Symons, A. B. Doser, D. F. Aldridge

Project Purpose

Reverse-time migration (RTM) is a novel wave propagation analysis technique that uses time-reversed seismic or acoustic signals to focus on the original energy source and image the earth's subsurface. RTM has numerous applications related to target identification and location (buried or above ground), CO_2 sequestration monitoring, geologic complexity estimation, as well as out-of-the-box applications such as ultrasonic nondestructive testing imaging and acoustic surgery.

RTM is accomplished by time reversing measured wave responses and using these signals to drive receiving transducers as sources, either using a numerical code or experimentally in the laboratory. The energy from receivers focuses at the source location at $t = 0$; if some of the energy is scattered, then it will focus at the scatterers for $t > 0$.

The objectives of this project are to take RTM from an intellectual curiosity to a working methodology for source characterization and imaging and to attach meaning to the images for the determination of material properties determining earth/material properties. Our approach is to understand the reverse-time process and determine the appropriate imaging condition for each application. In addition, our approach is to determine the role of eigenvectors for image processing and characterization of targets.

FY 2006 Accomplishments

Our key accomplishment is the successful production of images of simulated data, both for reflection data to image deep-earth structures, and surface waves used to image the near-surface, such as tunnels. Using simulated data, we produced an image of a step fault beneath a high-velocity pod, showing it is possible to image beneath this high-velocity occurrence. We produced images of shallow tunnels using simulated data. For certain types of seismic surveys, we quantified images to produce earth properties. Using simulated acoustic data, we showed that we can image an above-ground source when the line of sight to the source is limited. We are investigating eigenvector decomposition of the RTM images and the role of specific eigenvectors on image features. For certain applications, we developed a Shannon entropy method to produce RTM images.

Significance

RTM has potential for defense-related activities to include imaging cross-border tunnels, imaging hard and deeply buried targets, and battlefield management. RTM also has application to the oil and gas industries; for example, RTM can image beneath high-velocity subsurface salt intrusions as found in the Gulf of Mexico, whereas other types of migration algorithms fail to properly image beneath the salt. Subsalt imaging is important to the petroleum industry. RTM also can be used for imaging in nondestructive testing applications. This project has attracted interest from the Defense Advanced Research Projects Agency and from industry.

Refereed Communications

M.M. Haney, L.C. Bartel, D.F. Aldridge, and N.P. Symons, "Insight Into the Output of Reverse-Time Migration – What Do the Amplitudes Mean?," in *Proceedings of the 75th Inter. Mtg. Soc. Exploration Geophysicists*, pp. 1950-1954, November 2005.

Multispectral Detection of Microfluidic Separation Products

79751

C. C. Hayden, R. Meagher, C. C. Gradinaru, D. W. Chandler, A. K. Singh

Project Purpose

With the technology for microanalytical devices pushing toward smaller sample volumes and concentrations, there is an increasing need for highly selective detection of biomolecules (pathogens, toxins, or biomarkers such as cytokines) at very low concentrations. One path to more selective detection is to take advantage of specific interactions of fluorophore labels with their hosts that alter the fluorescence properties of the fluorophores. To this end, we have recently developed a new detection system used with a scanning confocal microscope to study biological systems at the molecular level. This detection system has the demonstrated sensitivity to record the full fluorescence spectrum and lifetime of individual molecules on a millisecond time scale.

In this project, we are combining microfluidic separation techniques with our lifetime- and wavelength-resolved detection capability to create a platform for unique scientific studies that will be a differentiating scientific capability for Sandia. In a single instrument, fluorescently tagged chemical complexes will be isolated by the microfluidic separation and analyzed by the multiplexed detection system, providing a high-throughput scientific tool for detailed studies of a variety of chemical processes in liquids, such as mobility, aggregation, ligand binding, and conformational fluctuations. Particularly powerful will be the unprecedented ability to examine the conformations and binding of novel biomolecular systems without immobilization.

Supported by these scientific studies, we are using this approach to develop a new generation of detection capabilities for microfluidic separation systems to detect bioagents or biomarkers of a disease with improved sensitivity (2-3 orders more sensitive than traditional fluorescence detectors) and multiplexing capability. The multispectral detection enables the simultaneous measurement of multiple analytes by using multiple fluorophores to selectively label species of interest. This approach has the opportunity to tremendously reduce false-positive results.

FY 2006 Accomplishments

The objectives of this work are to develop a unique scientific tool for studying chemical processes at the single molecule level while, at the same time, providing enhanced capabilities for multiplexed, ultrasensitive separations and immunoassays. The detection of individual molecules reveals fluctuations in molecular conformations and allows detailed studies of reaction kinetics such as ligand or antibody binding. These processes are revealed through their effects on the fluorescence properties of fluorophores detected by our new apparatus. The microfluidic technology offers unprecedented control of the chemical environment and flow conditions and affords the unique opportunity to study biomolecules without immobilization.

The detection system for the confocal microscope we developed for these studies enables us to record the wavelength, emission time relative to the excitation pulse, and the absolute emission time for each detected fluorescence photon with single molecule sensitivity. We used this capability to make fluorescence correlation measurements that show the binding of fluorescently-labeled ligands by proteins. Fluorescence spectra and lifetimes measured simultaneously with the fluorescence correlation show, for example, a large change in the lifetime of tetramethylrhodamine on biocytin when this ligand is bound by streptavidin protein. Analysis of this data at the single molecule level will reveal whether this quenching is a static process or varies as the fluorophore changes position relative to the protein.

We began similar studies on another ligand binding protein, maltose binding protein, which has a large conformational change upon binding. Using site-selective mutagenesis, we prepared protein mutants that can be labeled at specific sites where dye fluorescence properties will sensitively indicate the conformational changes. We also demonstrated the detection of analytes from electrophoresis on a microfluidic column with simultaneous measurement of fluorescence intensity, spectrum, and lifetime. This development provides new opportunities for sensitive and specific microfluidic analysis.

Significance

The field of single molecule studies is rapidly expanding, largely due to its potential for detailed studies of the chemistry of biological processes. A distinguishing characteristic of the chemistry of biological molecules is that their conformation, in conjunction with their chemical composition, creates distinct local chemical environments, such as binding sites, that govern many of their important reactions. Another important characteristic of biological molecules is that they frequently have multiple accessible conformations, so their local chemical environments (and, hence, chemical properties) vary both with time and from molecule to molecule within a sample.

Thus, for example, the reactivity or function of a biomolecule can vary from place to place within a cell. These variations in properties between molecules and the dynamics of their reactivity changes are often obscured due to averaging in measurements on ensembles of molecules. The promise of single-molecule studies is that they can reveal the detailed sequence of events involved in the reactions of complex molecules and measure the difference in properties between molecules. A critical problem in single-molecule studies is the difficultly in making measurements without perturbing the molecule's activity due to the need to immobilize the molecules.

In this project, we are developing microfluidic methods for single-molecule studies that do not require immobilization of the molecules of interest and yet allow precise control of concentrations and reactant mixing times. This capability will enable studies of a much wider range of biomolecules and processes than previously possible. Results from the work on this project contributed to a proposal we submitted for a broad program in this area, which was funded by DOE Basic Energy Sciences.

The research in this project takes advantage of a time-resolved multispectral single-molecule detection system we developed. The combination of microfluidic devices and this detection system with single-molecule sensitivity is also being used to develop new methods for detection and identification of analytes in microfluidic devices. The capability for simultaneous detection of multiple fluorescence properties can enhance the certainty in identification of analytes and single-molecule detection takes sensitivity to a new level. The methods we are developing should impact many application areas for microfluidic reaction and separation systems.

Other Communications

C.C. Hayden, "Time-Resolved Multispectral Single-Molecule Spectroscopy," presented at 53rd Annual Western Spectroscopy Association Conference, Pacific Grove, CA, February 2006.

Interface Physics in Microporous Media

93496

J. T. Fredrich, M. A. Hickner, D. R. Noble, D. J. Holdych

Project Purpose

The purpose of this project is to increase fundamental understanding of multiphase transport through microporous media. One challenge relates to the extraordinary geometric and topologic complexity of the pore space of microporous media. The past decade has seen the advancement of high-resolution three-dimensional (3D) imaging techniques that, when coupled with new numerical simulation techniques, enabled a step change in our ability to accurately model single-phase fluid flows in microporous media. Multiphase flows, however, are intrinsically more complicated to analyze and model due to the existence of fluid-fluid interfaces that can carry nonzero stresses.

The relative phase saturation is not sufficient to describe the macroscopic state of the system, which instead is affected by the geometry of the interfaces between the fluid phases and between the fluid and solid phases. However, quantification of interfacial areas has eluded direct measurement, which in turn has hampered advancement in the field of multiphase flows and, thus, development of predictive capabilities. This project will produce a breakthrough in our ability to fundamentally understand and quantitatively predict multiphase flows in microporous media.

A key aspect of the research is coupling between fundamental experimental discovery and synergistic development of modeling and simulation tools. The lattice Boltzmann (LB) method has been frequently employed for modeling fluid flows, and a primary advantage of the method includes the ability to accommodate complex morphologies. The method also lends itself toward implementation on massively parallel architectures – a key advantage for the problems of interest here. The primary difficulties with modeling multiphase flows in microporous geometries with LB are twofold.

Thus far, all multiphase LB simulations have used a diffuse interface approach that smears the interfacial physics over a distance of several lattice widths. This effect may be acceptable if this diffuse interface width is much smaller than the characteristic size of the geometry. For flow in micropores, however, this smearing might occur over the entire pore, so a sharp interface approach is required to accurately capture the physics of multiphase flow in microporous systems.

A second problem with current LB approaches is the limit on density and viscosity ratios, which are typically limited to less than 5:1. This strongly constrains the types of microfluidic systems that can be simulated with current LB technology.

In this project, we are developing new LB methods that implement sharp interface technology developed in the context of level set methods. This technology will allow simulation of the sharp interfacial physics together with the true density and viscosity ratios. Direct comparisons of numerical predictions versus experimental observations should be possible from the micromodel experiments and also from macroscopic laboratory-scale measurements of multiphase flow. The data we acquire will enable direct testing of theories and models of the role of interfacial physics in multiphase fluid dynamics.

FY 2006 Accomplishments

- Placement of contracts with university collaborators
- Proof-of-concept of proposed two-photon lithographic technique for fabricating 3D micromodels
- Design and construction of dedicated two-photon laser system for photolithography
- Development of procedures to optimize fabrication of 3D micromodels
- Identification of key remaining experimental issues

- Fabrication of two 3D micromodels
- Fabrication of two-dimensional (2D) micromodels based on image data and random porous media
- Design of 2D micromodels for in situ miscible flow experiments
- Fabrication of 2D micromodel for in situ miscible flow experiments
- Design of experimental apparatus for in situ multichannel laser scanning confocal microscopy multiphase flow experiments
- In situ multichannel laser scanning confocal microscopy multiphase flow experiments to develop experimental procedures and optimize imaging parameters
- New input/output routines written for 3D image analysis software
- Successful proposal for beamline time at the Advanced Photon Source (APS) at Argonne National Laboratory
- Successful 3D synchrotron microtomographic experiments conducted at APS
- Development of a new, efficient solution method for lattice Boltzmann simulation of transport in microporous media

Significance

The new LB solution algorithm developed is of interest to the general scientific community because it reduces solution time. Ultimately, the development of experimental and numerical simulation capabilities in multiphase flow will produce a breakthrough in our ability to understand and predict the behavior of engineered and natural microfluidic systems in the chemical, earth, engineering, physical, and biological sciences. Potential applications include µChemLab™, microelectromechanical systems (MEMS), microporous fuel cell components, membrane and filtration technology, porous ceramics, chromatography, oil and gas reservoirs, and aquifers.

Refereed Communications

D. Holdych, "Direct Simulation of Idealized Tendon Tissue with Lattice Boltzmann," presented at 15th Discrete Simulation of Fluid Dynamics, Geneva, Switzerland, August 2006.

D.R. Noble, "Full Newton Lattice Boltzmann Method for Time-Steady Flows," presented at 15th Discrete Simulation of Fluid Dynamics, Geneva, Switzerland, August 2006.

Creating a Discovery Platform for Defined-Space Chemistry and Materials: Metal-Organic Frameworks

93497

M. D. Allendorf, J. W. Hsu, J. A. Greathouse, B. A. Simmons

Project Purpose

Metal-organic frameworks (MOF) are a recently discovered class of nanoporous, defect-free crystalline materials. MOFs have tunable, monolithic pore sizes and cavity properties due to their structure and crystalline nature, enabling rational synthetic design of porous materials at the molecular level. MOFs have properties exceeding those of most other porous materials, including the lowest known density, highest surface area, tunable photoluminescence, highly selective molecular adsorption, and methane sorption rivaling gas cylinders. These unusual properties are achieved by coupling inorganic metal clusters with organic ligands that serve as struts, allowing facile manipulation of pore size and surface area through reactant selection.

The objectives of this project are to 1) establish MOFs as a discovery platform for exploring and understanding confined-space chemistry and 2) to enable the exploitation of their unique properties by creating, for the first time, MOF thin films on a range of useful substrates. To achieve these objectives we are applying and expanding Sandia's expertise in interfacial chemistry, multiscale modeling, and material systems integration.

The project comprises three tasks. In Task 1 we are synthesizing MOFs with tunable pore size and chemistry, characterizing their properties, and using this knowledge to identify suites of MOFs to address key Sandia missions. In Task 2, we are validating and developing robust models that reveal the links between pore electronic structure and macroscopic observables such as molecular adsorption. In Task 3, we are developing methods to create MOF layers on semiconductors, optical materials, and plastics, and exploring simple device concepts to assess the potential for MOFs to serve as sensors and separations media.

FY 2006 Accomplishments

We synthesized the first-ever photoluminescent MOF based on a zinc-stilbene metal-linker combination. This material is an ultrahigh surface area, three-dimensional cubic structure. The presence of luminescence shifts upon guest absorption provides a potential sensing transduction mechanism.

We developed the first nonbonded forcefield to describe a MOF, enabling atomistic simulations of chemical reactions and sorption processes that affect the MOF structure. The forcefield was used to model the interaction of water molecules with zinc MOFs using molecular dynamics simulations. In agreement with experiment, we showed that the MOF structure collapses when the amount of water absorbed exceeds a certain limit.

We grew thin films of zinc MOFs on self-assembled monolayers (SAM) and anodized aluminum oxide (AAO). We improved the method of growing MOFs on SAMs and created MOF-on-AAO materials for testing as gas separation membranes.

Through a collaboration with Washington State University, we performed the first-ever measurements of MOF mechanical properties, yielding elastic modulus values in excellent agreement with ab initio predictions.

We established three new collaborations: 1) with Prof. David Bahr, Washington State University, for nanoindentation measurements; 2) with Prof. Roland Fischer, Ruhr-Universitat Bochum, Germany, for MOF synthesis; and 3) with Prof. T. Timofeeva, New Mexico Highlands University, for single-crystal x-ray diffraction.

We hosted a graduate student from Prof. Fischer's group, enabling us to establish full MOF synthesis capability in less than two months.

Through interactions with other Sandia staff, we established new analytical and theoretical capabilities for MOFs: single-crystal fluorescence microscopy, electrical measurements, fluorescence lifetimes, and plane-wave density functional theory (DFT).

Significance

MOFs provide a controllable, defect-free model for chemistry within confined spaces, as well as a high surface area material with uniform and controllable properties that have numerous device applications. If successful, we expect immediate impact on molecular trapping/ion exchange for water or waste stream cleanup and chemical weapons detection. Other potential impacts include 1) transport phenomena in porous media (relevant to nuclear waste storage); 2) gas sorption, including the problems of carbon dioxide sequestration and reversible storage of reactive gases in microdevices; 3) separation methods, such as size exclusion chromatography, for removal of small molecules from streams containing larger biological compounds; and 4) permeable membranes for fuel cells.

Refereed Communications

J.A. Greathouse and M.D. Allendorf, “Reactivity of Metal-Organic Framework-5 with Water Studied by Molecular Dynamics Simulations,” *J. Amer. Chem. Soc.*, vol. 128, p. 10678-10679, August 2006.

J.A. Greathouse and M.D. Allendorf, “Molecular Dynamics Simulations of Metal-Organic Framework-5 and Its Interactions with Water,” presented at American Chemical Society Fall Meeting, San Francisco, CA, September 2006.

F. Schroeder, S. Hermes, A. Baunemann, C. Bauer, A.J. Skulan, B.A. Simmons, M.D. Allendorf, C. Wöll, and R.A. Fischer, “Thin Films of Metal-Organic Framework Compounds: Design and Characterization of New Functional Surfaces,” presented at American Chemical Society Spring Meeting, Atlanta, GA, March 2006.

C. Bauer, F. Schröder, A.J. Skulan, A.A. Talin, R. Anderson, R.A. Fischer, B.A. Simmons, and M.D. Allendorf, “Electronic and Luminescent Properties of Metal-Organic Frameworks: Toward Gas Sensors,” presented at American Chemical Society Spring Meeting, Atlanta, GA, March 2006.

J.A. Greathouse, R. Schmid, and M.D. Allendorf, “Development of a Nonbonded Forcefield for Molecular Modeling of Metal-Organic Frameworks,” presented at American Chemical Society Spring Meeting, Atlanta, GA, March 2006.

A.J. Skulan, C. Bauer, F. Schroeder, S. Hermes, R.A. Fischer, B.A. Simmons, and M.D. Allendorf, “Hybrid Materials of Metal-Organic Frameworks (MOF),” presented at 209th Meeting of The Electrochemical Society, Denver, CO, May 2006.

Computational & Information Sciences

Massively Parallel Scalable Atmosphere Model

67015

W. F. Spotz, M. A. Taylor, R. C. Schmidt, M. B. Boslough

Project Purpose

The purpose of this project is to demonstrate scalable performance of a global atmospheric model on massively parallel computer architectures. Reliable climate modeling is an increasingly important capability for social, economic, and security policy planning. It is also one of the most computationally intensive applications in science.

Of the different components that comprise a climate system model (atmosphere, ocean, sea-ice, and land, plus the coupler that ties them together), the atmosphere consumes the greatest computational resources. Traditional atmosphere models based on the spectral transform method have always vectorized well, albeit with a somewhat limited granularity. On more general and less expensive distributed-memory systems, this type of atmosphere model does not parallelize well, for several reasons: expensive all-to-all transposes of data; Legendre transforms which do not scale linearly; and the aforementioned granularity issue.

We approached the scalability issue by collaborating with the National Center for Atmospheric Research (NCAR) on a spectral element atmospheric model. In this method, the surface of the earth is tiled with quadrilaterals, each of which is a high-order finite element (typically 8 by 8). These elements define locally supported basis functions that eliminate the need for expensive data transposes, replacing them with efficient edge communications.

The Legendre transforms are replaced with scalable, piecewise polynomial computations, and the granularity limitation is greatly reduced because the problem can be decomposed down to one element per processor. We knew the scalability properties of the spectral element method before the start of the project.

The objectives of the project were to demonstrate the model's capabilities at higher resolutions and at larger processor counts than had been achieved before, and to help mature the model so that it could be adopted by the climate modeling community as a component within the Community Climate System Model (CCSM), a production-quality climate model maintained at NCAR. This will allow the advantages of the spectral element dynamical core to be coupled to existing atmospheric physics parameterizations, ocean dynamical cores and physics parameterizations, and sea-ice and land models, so that climatologists can better take advantage of the terascale computers of today and the petascale computers of tomorrow.

FY 2006 Accomplishments

We demonstrated a high degree of scalability for explicit time stepping, maintaining approximately 80 percent efficiency on over 8,000 processors on Red Storm, achieving 5 teraflops. On Blue Gene/L, which has a larger number of slower processors, parallel efficiency stayed above 50 percent for 32,000 processors for the 10 km case, the highest resolution we ran. The maximum flop rate achieved on Blue Gene/L was 4 teraflops.

We completed three demonstration runs on Red Storm: an atmospheric simulation of the polar vortex problem with the spectral element model, and two 10-year simulations using POP, the Parallel Ocean Program from Los Alamos National Laboratory. The polar vortex run consisted of 1 billion grid points (13 km spacing in the horizontal and 300 vertical levels), which ran for 288,000 time steps on 7200 processors for 36 hours, producing 1 terabyte of output. The POP simulations were 350 million grid points (1/10 of a degree) on 5000 processors, producing 1.5 terabytes of output. All of these simulations were at higher resolutions than had previously been executed, and the ocean simulations captured phenomena never previously captured by a global model.

We also improved semi-implicit time-stepping capability, which increases the allowable time step but adds the expense of a Helmholtz problem to solve. We can accelerate the overall integration rates (simulated time per wall clock time) compared to explicit by a factor of two to three for moderate processor counts (1000 processors or less), although this acceleration drops off for higher processor counts.

A major problem we faced was coupling the spectral element model to the community atmosphere model (CAM). CAM is the atmospheric component of CCSM, and it is CAM that will provide a large set of atmospheric physics parameterizations that come from a wide diversity of disciplines: solar radiation, cloud physics and precipitation, aerosols, chemistry, and so on.

Our early experience is that this coupling leads to numerical ringing, a high degree of artificial oscillations in the solutions that need to be damped out. The traditional spectral transform method filters these out "naturally" because it employs the two-thirds truncation rule to prevent aliasing error, and this truncation also suppresses induced oscillations from the physics parameterizations. Another approach in the realm of atmospheric models is the finite volume method, which can employ flux limiters to preserve monotonicity and local conservation.

The spectral element model cannot employ a global truncation because it is a local method, nor can it employ flux limiters. Instead, we employ a hyperviscosity filter that effectively damps out forcings that occur on a scale too fine for the grid to accurately represent.

As a result of the work done in FY 2006, we have a viable hyperviscosity filter for the spectral element atmosphere model, a crucial step in coupling to CAM and CCSM.

Significance

Climate models have improved a great deal in the last 30 years, but the challenge of accurately predicting the earth's climate for perhaps the next 100 years under a variety of scenarios remains a challenging one. Predicting regional climate change is even more difficult, but necessary for policy makers. There are two general areas where improvements to climate models (and the CCSM in particular) can be made: increasing resolution, and improving subgrid physics parameterizations. This project addresses the first area.

Doubling the spatial resolution results in an increase in computation time of a factor between 10 and 12 (two horizontal directions for a factor of four, the time step halves for stability thus doubling the computations to eight, and typically the vertical resolution is increased more slowly, perhaps the square root of two for a total increase between 10 and 12). Clearly, to support such an increase, we need faster computers. But we also need efficient numerical schemes that maintain that efficiency when thousands of processors are applied. We demonstrated that the spectral element method is just such a scheme.

In a follow-on project, as part of the SciDAC (Scientific Discovery through Advanced Computing) Climate Consortium, Sandia will help integrate the spectral element method into CCSM. When completed, climatologists will be able to use the computers they currently use much more efficiently and be able to use much larger machines than they were able to use before. This in turn will allow longer simulations, higher resolution simulations, and ensembles with a greater numbers of simulations, all of which will lead to a better understanding of how the earth's climate may change in the future and provide policy makers with better data upon which to base their decisions.

Refereed Communications

B.T. Nadiga, M.A. Taylor, and J. Lorenz, "Ocean Modeling for Climate Studies: Eliminating Short Time Scales in Long-Term, High-Resolution Studies of Ocean Circulation," to be published in *Mathematical Computer Modeling*.

F.X. Giraldo and M.A. Taylor, "A Diagonal Mass Matrix Triangular Spectral Element Method Based on Cubature Points," to be published in *Journal of Engineering Mathematics*.

B.A. Wingate and M.A. Taylor, "Measuring Characteristic Length Scales of Prolate Spheroidal Wave Functions in One and Two Dimensions," submitted to *Journal of Engineering Mathematics*.

J. Dennis, A. Fournier, W.F. Spotz, A. St-Cyr, M. Taylor, S. Thomas, and H. Tufo, "High-Resolution Mesh Convergence Properties and Parallel Efficiency of a Spectral Element Atmospheric Dynamical Core," *The International Journal of High Performance Computing Applications*, vol. 19, pp. 225-235, Fall 2005.

B.A. Wingate and M.A. Taylor, "Performance of Numerically Computed Quadrature Points," submitted to *Applied Numerical Mathematics*.

M.A. Taylor and B.A Wingate, "A Generalization of Prolate Spheroidal Functions with More Uniform Resolution to the Triangle," to be published in *Journal of Engineering Mathematics*.

M.A. Taylor, B.A. Wingate, and L.P. Bos, "A Cardinal Function Algorithm for Computing Multivariate Quadrature Points," submitted to *SIAM Journal on Numerical Analysis*.

Other Communications

W.F. Spotz and M.A. Taylor, "Massively Parallel Performance of HOMME," presented at the 2006 Workshop on the Solution of Partial Differential Equations on the Sphere, Monterey, CA, June 2006.

W.F. Spotz and M.A. Taylor, "Massively Parallel Performance of the HOMME Spectral Element Atmosphere Model," presented at the 8th International Workshop on Next-Generation Climate Models for Advanced High-Performance Computing Facilities, Albuquerque, NM, February 2006.

W.F. Spotz and M.A. Taylor, "Advances in Climate Modeling with High-Performance Computing," presented at the Sandia Earth Day Celebration, Albuquerque, NM, April 2006.

High-Performance Processing Architecture

67016
K. S. Hemmert, K. D. Underwood

Project Purpose

For the last few decades, Moore's law has led to higher clock speeds and larger numbers of transistors on microprocessors. However, memory technology has not improved as rapidly ("Von Neumann bottleneck" and "memory wall" are terms commonly used to refer to different aspects of this problem). Computer architects have been forced to dedicate most of the transistors on a chip to compensate for this problem. These efforts cause processors to draw more power, yet processors still fail to sustain peak performance.

Reconfigurable computing is a revolutionary technology that may better use the available transistors to achieve higher density computing and a higher fraction of peak performance. Unfortunately, it faces a number of constraints that may limit its applicability to Sandia applications. We are studying the performance of reconfigurable computing on Sandia's scientific applications. Specifically, we are studying the subset of reconfigurable computing that is based on field-programmable gate arrays (FPGAs).

While reconfigurable computing (using FPGAs) may hold keys to future high-end computing, there are significant hurdles to its deployment. Changes to the programming model will be necessary, and applications could require significant recoding efforts. Thus, it is important that we fully understand the performance implications of such a move.

There are two aspects of an FPGA-enhanced system that must be understood. First, we must determine the potential speedup of Sandia's applications using an FPGA by exploring what parts of the application are amenable to mapping onto an FPGA and then determining what the performance of the application will be if that part is remapped onto an FPGA. Second, we must determine the correct architecture of an FPGA-enhanced supercomputer. In the near future, this translates to determining the architecture of the nodes of a massively parallel processing supercomputer.

It is not obvious how an FPGA can be integrated into a supercomputer node without disrupting system balance. A longer-term view requires determining how, or if, a complete supercomputer could be built based on future FPGA devices.

FY 2006 Accomplishments

We primarily focused on the question of how FPGAs should be integrated into systems. In support of this, we took measurements from the Cray XD1 system and built a system-level simulator that modeled the behavior encountered with the XD1. This enabled us to vary system-level parameters to define requirements for future FPGA-based systems in terms of system-level latencies and bandwidths.

We also continued work on floating-point units for FPGAs. These units are widely recognized as the best available and are currently in use at Los Alamos National Laboratory, the California Institute of Technology, the University of Cincinnati, and others.

A third aspect of this year's work involved a collaboration with the University of Washington in which we explored architectural modifications to further enhance floating-point performance on FPGAs.

Significance

Large-scale simulation is a fundamental tool in the fields of science, energy, and defense. As such, DOE is now dependent on supercomputers to fulfill its mission. Currently, those supercomputers are based upon commodity microprocessors, but many researchers inside and outside of DOE foresee limitations of that model that could impact the DOE mission within a decade. FPGAs could be a leading candidate for a radical shift in the fundamental architecture of supercomputers.

FPGAs have been increasing in performance faster than commodity processors. As a result, FPGAs have the potential to exceed the peak floating-point performance of microprocessors within five years. More importantly, there are indications that FPGAs may be able to sustain a higher percentage of peak performance than microprocessors. If this trend continues, the sustainable performance of an FPGA may soon dramatically exceed the sustainable performance of a microprocessor. As such, FPGAs may become a key component of future high-performance computing systems. However, it is not clear how, or if, that performance can be harnessed for Sandia applications.

Before Sandia makes a major investment to incorporate FPGAs into future supercomputing systems, it is critical to determine whether FPGAs will provide a significant performance improvement for Sandia applications. Just as importantly, we must determine how hundreds or thousands of FPGAs should be integrated into an MPP supercomputer. Knowing that an FPGA can accelerate Sandia applications is not enough. If integrated into the system incorrectly, that acceleration could be negated.

Considering the two-year development cycle of many supercomputers and the five-year time horizon until FPGAs outperform commodity processors, it is time to address this issue now. The modeling performed as part of this project will form a basis for understanding how systems incorporating FPGAs might perform. More importantly, it will answer questions about the best way to integrate FPGAs into the system. Because FPGAs are becoming more common offerings on high-performance computing platforms, it is important for DOE to explore this technology. In addition, FPGAs share many properties with accelerators in general, which make them a useful platform for studying accelerator issues.

Refereed Communications

K.D. Underwood, K.S. Hemmert, and C.D. Ulmer, “Architectures and APIs: Assessing Requirements for Delivering FPGA Performance to Applications,” in *Proceedings of the 2006 ACM/IEEE Conference on Supercomputing*, p. 111, November 2006.

M.J. Beauchamp, S. Hauck, K.S. Hemmert, and K.D. Underwood, “Architectural Modifications to Improve Floating-Point Unit Efficiency in FPGAs,” in *Proceedings of the IEEE International Conference on Field Programmable Logic and Applications*, pp. 515-520, August 2006.

M.J. Beauchamp, S. Hauck, K.D. Underwood, and K.S. Hemmert, “Embedded Floating-Point Units in FPGAs,” in *Proceedings of the ACM/SIGDA Fourteenth ACM International Symposium on Field-Programmable Gate Arrays*, pp. 12-20, February 2006.

Substructured Multibody Molecular Dynamics

67017
P. S. Crozier, M. J. Stevens, S. J. Plimpton

Project Purpose

The purpose of this project is to develop advanced molecular simulation capabilities for our LAMMPS (large-scale atomic/molecular massively parallel simulator) software, especially the ability to perform substructured multibody molecular dynamics (SMMD) simulations. Specific goals include enabling the following:

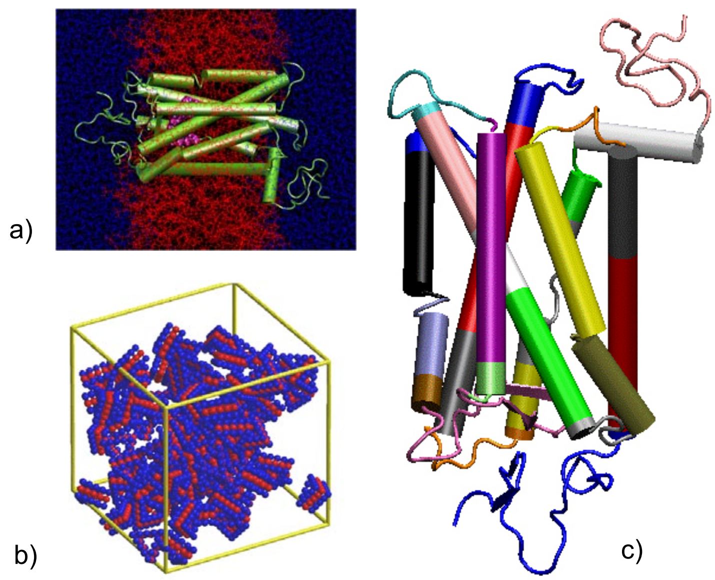
- bigger/longer/faster molecular dynamics (MD) simulation of complex nano/bio systems of interest
- coupled rigid-body simulation
- seamless integration of systems with disparate inherent timescales within MD framework
- faster simulation through removal of nonessential degrees of freedom
- automatic substructuring of systems into fast/medium/slow/zero motion regions.

Other objectives include improved implicit solvent options available within LAMMPS and testing and demonstration of the code developments with meaningful applications.

Specific tasks that contribute to these goals include development of a force-matching algorithm that uses atomistic force-field simulations to parameterize coarse-grained force-field parameters for much faster simulation within the SMMD framework, completion of the coupling between an articulated rigid-body simulation code and LAMMPS, and automation of time-step size selection and substructuring.

FY 2006 Accomplishments

We developed software that allows users to perform coupled rigid-body simulations within a molecular dynamics framework. The Rensselaer Polytechnic Institute software POEMS (parallelizable open source efficient multibody software) was integrated into Sandia's LAMMPS molecular dynamics simulation package.



a) 150-ns biomolecular simulation of rhodopsin photoisomerization shows early steps in visual sensing.
b) Simulation of functionalized nanoparticle self-assembly.
c) Seamless multigranular substructured molecular dynamics now possible using Sandia's LAMMPS code.

A new LAMMPS module, "fix poems," was written to enable communication between LAMMPS and POEMS. LAMMPS computes forces and torques on each rigid body and sends that information to POEMS at each time step. POEMS integrates the coupled rigid body equations of motion for each system of coupled rigid bodies, and returns updated position and momentum information to LAMMPS. Users can now perform articulated rigid-body dynamics simulations of complex nano- and biomaterials within LAMMPS.

We investigated several test problems to ensure that the new articulated rigid-body dynamics capability was working properly. We demonstrated that our enhanced software produced correct thermodynamic, kinetic, and structural characteristics for a simple alanine dipeptide test problem using both an atomistic and rigid-body model. Other test problems, including alkane systems, a RuBisCO protein system, a rhodopsin protein system, and a nanoparticle self-assembly system likewise yielded positive results confirming the reliability of the newly developed capabilities.

Significance

At the conclusion of this project, the tools are in place to perform coupled rigid-body simulation within the molecular dynamics framework. Future molecular simulation efforts, especially biomolecular and nanoparticle simulation efforts, can now take advantage of these unique capabilities to accelerate their simulations and make possible simulations that would otherwise be impossible due to computational constraints.

With the recent commencement of the Center for Integrated Nanotechnology and an increase in biotechnology research here at Sandia, such simulations will play an increasingly important role in discovering and understanding physical phenomena from the molecular level up. The simulation tools developed during this project make it easy for researchers to perform rigid-body dynamics simulations important to nanoparticle self-assembly, protein dynamics, and many other nano- and biomolecular materials investigations.

Enhancing Simulation Performance on Clusters with Configurable Auxiliary Devices

67018

C. D. Ulmer, M. L. Leininger, D. C. Thompson, R. C. Armstrong

Project Purpose

Current generation high-performance computing (HPC) systems rely almost exclusively on general-purpose processors to perform the complex calculations needed by today's scientific computing applications. While readily available and easy to program, these processors typically operate far below their peak performance levels due to software overhead.

One option for obtaining better performance is to implement an algorithm in custom-built hardware that maximizes processing concurrency. For example, application-specific integrated circuits (ASICs) have been constructed to greatly accelerate molecular dynamics computations. However, ASICs are extremely expensive to develop and are challenging to adapt to other application domains.

Reconfigurable computing (RC) is a more practical approach to hardware acceleration that leverages reconfigurable hardware devices such as field-programmable gate arrays (FPGAs). In addition to being capable of emulating large computational circuits, FPGAs can be reprogrammed as needed by the user. Recent advances in commercial FPGA capabilities present an opportunity to exploit these devices as economical "soft ASICs" that can be used to accelerate scientific calculations.

The purpose of this project is to investigate how RC techniques can improve the performance of scientific applications that are relevant to Sandia. Our effort focuses on two key issues that are essential for making RC practical.

First, we examine the system integration issue: how can FPGA resources be added to a modern HPC platform in a manner that maximizes performance and minimizes the disruption of the system architecture? For this work we consider the trade-offs between 1) HPC systems that feature built-in FPGA accelerators and 2) a networked approach where FPGAs are connected to the communication network of an existing HPC system.

Second, we examine the adaptation issue: what design techniques enable an FPGA to achieve performance gains in real applications on real systems? For this work we have adapted multiple, nontrivial computational kernels to FPGA hardware in order to gain a better understanding of 1) the characteristics of applications that map well to hardware and 2) RC techniques that can be exploited to achieve performance gains.

Our work leverages and complements the work being performed in the "High Performance Processing Architecture" LDRD project (67016), which is addressing the design of high-precision floating-point units for FPGAs.

FY 2006 Accomplishments

In the final year of this project, we concentrated on adapting a broad range of nontrivial computational kernels to FPGA hardware. We adapted six different kernels to hardware using a variety of design techniques. Each of these computational kernels employs multiple (3-50) floating-point units in a single-chip FPGA accelerator. We measured performance using the Cray XD1 HPC platform, which tightly couples Xilinx Virtex II/Pro (V2P) FPGAs with AMD Opteron processors.

The first two computational kernels we constructed this year were built in collaboration with the "High-Performance Processing Architecture" LDRD project team. We constructed FPGA designs for both double-precision general matrix multiply (DGEMM) and fast Fourier transform (FFT) algorithms. While memory bandwidth bottlenecks in the XD1 prevented the DGEMM FPGA design from obtaining a performance gain, the FFT FPGA design achieved an 8x performance gain over an AMD Opteron processor. This work exposed the need to employ a nonblocking interface when using the FPGA as a coprocessor in order to hide data transfer overheads.

Based on the lessons of the DGEMM and FFT designs, the next two algorithms (STIFF and kNN) that we adapted to hardware employed large data sets that enabled us to better pipeline the flow of data between the host and the FPGA accelerator.

In STIFF we achieved a 4x performance gain with the FPGA for an algorithm that computes the physical stiffness property of each node in a mesh. The STIFF hardware design reuses floating-point units to perform different operations in the algorithm, and processes multiple mesh nodes concurrently in order to improve performance.

In kNN we achieved up to a 7x performance gain in locating the k-nearest neighbors to an input vector in a training data set. The kNN design loads a large amount of training data into memory located in close proximity to the FPGA and employs eight Euclidean distance engines to compare multiple vectors at a time.

The final two computational kernels (RTI and ISO) we constructed this year perform operations frequently required in visualization. In the ray-triangle intersection (RTI) design, we constructed FPGA hardware that computes the point at which a ray intersects a triangle. When used in a photon mapping application where it is necessary to compute the intersection points between many rays and many triangles, the FPGA design achieved an 11x performance gain over software.

Finally, in the isosurfacing (ISO) design, we constructed parallel hardware to rapidly perform a large number of threshold comparisons on a large volume of data in order to generate a polygon representation of the isosurface. Based on the characteristics of the input data set, the FPGA accelerator achieved a 4-30x performance gain over the host processor.

Significance

A number of conclusions can be drawn from the work performed in this project. First and foremost, we confirmed that FPGAs can be used to accelerate the performance of certain scientific computing applications under the proper circumstances. However, performance gains are largely dependent on multiple factors, including the characteristics and partitioning of the algorithm, the features of the FPGA accelerator, and the quality of the hardware adaptation effort.

From our experiences, we believe that a successful FPGA accelerator must 1) be tightly coupled with the host processor, 2) use a high-capacity FPGA, and 3) provide a moderate amount of wide, local memory. Both the Cray XD1 and the SRC-7 illustrate these properties.

In terms of commercial FPGAs, our work demonstrates that current generation FPGAs provide enough capacity to house small but interesting scientific computations. As illustrated in our RTI design, a medium-sized FPGA such as the V2P50 can house over 50 floating-point units. By chaining units together in a deep pipeline, it is possible to maximize concurrency and achieve sizable performance gains.

While it is possible to house even larger designs by using multiFPGA boards, our belief is that it is more advantageous to spread the FPGAs among different HPC nodes and attack smaller computations with the individual FPGAs.

Beyond traditional HPC applications, this work will benefit other Sandia applications where it is necessary to place a large amount of compute capability in the field. FPGAs offer dense computing platforms with excellent power consumption characteristics. Additionally, FPGAs are attractive in real-time applications because the designer has fine-grained control over the timing of computational events within the FPGA.

Based on these characteristics, we believe that FPGAs represent a pivotal technology for enabling next-generation defense and intelligence applications.

As users demand more and more processing in their deployable systems, there are few technologies other than RC that will be capable of meeting the stringent size, power, and processing demands of the applications. As such, we believe that the work in this LDRD project is important to a variety of emerging applications.

Refereed Communications

C.D. Ulmer and A. Javelo, “Floating-Point Reuse in an FPGA Implementation of a Ray-Triangle Intersection Algorithm,” in *Proceedings of the International Conference on Engineering of Reconfigurable Systems and Algorithms (ERSA)*, pp. 93-102, June 2006.

C.C. Clark, C.D. Ulmer, and D.E. Schimmel, “An FPGA-Based Network Intrusion Detection System with On-Chip Network Interfaces,” *International Journal of Electronics*, vol. 93, pp. 403-420, June 2006.

Penetrator Reliability Investigation and Design Exploration (PRIDE)

67020
M. L. Martinez-Canales, S. W. Thomas, G. A. Gray, R. Heaphy, L. P. Swiler, P. D. Hough, T. G. Trucano, M. L. Chiesa, R. R. Settgast, P. J. Williams

Project Purpose

The focus of our work was research and algorithmic development in optimization under uncertainty (OUU) problems arising in earth penetrator (EP) design. While EP design was our driving application, the challenges therein span the gamut of engineering applications.

With decreasing resources and a real inability to physically test parts of the design space, more and more engineering applications have been turning to modeling and simulation (M&S) as attractive alternatives to time-consuming hand-design and expensive field/laboratory testing. However, because conventional M&S approaches are also cost-prohibitive, even more so when the effects of uncertainty are fully accounted, in this project we investigated specific algorithms that would reduce the overall computational expense of the design process to enable the design of credible, higher-quality, reliable, and robust engineering designs.

To understand areas in which computational reductions could be made, we first sought to understand a typical engineering analysis process. Typically, one or more low-fidelity (simple geometry with a small number of engineering state and control variables), low-mesh-resolution (coarse mesh) models are initially constructed to explore the parameter/design space. Using the simpler models as surrogates of the complex system, the analyst culls from the surrogate model responses, at specific parameter/design points deemed interesting, a sense of how to construct, explore, and analyze subsequent higher-fidelity and higher-mesh-resolution models that will be computationally more expensive.

This process is sequential and requires multiple iterations until the analyst is satisfied with the outcome. Note that the cheaper models are built first because further model modifications can be rapidly incorporated. Unfortunately, the cheaper models may not represent the physics of the higher-fidelity system very well, which becomes problematic when design optimization is used. In design optimization, the optimization algorithm iterates across parameter/design configurations until it finds one that is best. If the structure of the higher-fidelity models is not represented by the lower-fidelity model, the result of a traditional design optimization algorithm, using the simpler model, will be misleading when applied to the truer high-fidelity systems.

In scoping our efforts, we concentrated on addressing three main challenges in the engineering analysis process while also taking into account the presence of uncertainty. The first challenge required leveraging small local samples, already constructed by optimization algorithms, to build effective surrogate models (FY 2004 efforts). The second challenge was to develop a methodical design process based on multiresolution, multifidelity models (FY 2005 - FY 2006 efforts). The third challenge involved the development of computationally feasible tools and the implementation of design processes useful to the weapons community and beyond (FY 2006 efforts).

FY 2006 Accomplishments

Test Problem Construction:
Our research test suite consisted of one engineering suite and one mathematics suite. The engineering suite consisted of a set of EP models of multiple fidelity and mesh resolution. We generated the models using Sandia software codes Cubit and Presto and parameterized the models by both design and uncertain variables. The mathematics suite consisted of a multidimensional analytical function with different characteristics (such as location and number of stationary points) at different dimensions.

Gaussian Process Models:
Our M&S process improvement approach involved first modeling deterministic computer output as realizations of a Gaussian process (GP). The set of GPs indexed by the simulation process parameters and their prior distributions then represent our prior uncertainty regarding possible output surfaces.

We used newly observed or additionally generated data to transform the prior distribution into a posterior distribution under the Bayesian paradigm. By using the cheaper GP as a surrogate of the complex model (GP-s), we achieved reasonable computational savings in parameter space exploration. We implemented our GP-s in Matlab and in DAKOTA. By using the GP as a corrector between multiple model fidelities (GP-c), not only did we observe a reduction in computation costs, but we also more closely emulated the engineering process. We also implemented GP-c in Matlab.

Optimization Under Uncertainty Algorithms:
To reduce the overall computing expenses associated with design optimization, we integrated GPs at both the "local" and "global" levels of optimization algorithms to construct new OUU algorithms: OUU-LGP and OUU-GGP, respectively.

In the OUU-LGP approach, we used the GPs in two different ways within the trust-region of a surrogate-based optimization method, creating two OUU-LGP variants. In the first approach, we used GP-s to create OUU-LGP-s. We successfully tested and implemented OUU-LGP-s in the DAKOTA framework, making use of both the existing trust-region (OPT++) surrogate-based optimization infrastructure and our GP implementation. In the second approach, we used GP-c to create OUU-LGP-c. To circumvent some DAKOTA infrastructure limitations associated with multilevel surrogate constructions, we implemented and successfully tested OUU-LGP-c in Matlab against our analytical test function.

In the OUU-GPP approach, we used a Treed GP (tgp) algorithm (developed by University of California at Santa Cruz collaborators Herb Lee, Robert Gramacy, and Matt Taddy) in a pattern search optimization algorithm (APPSPACK).

Within an Oracle module separate from the search pattern, the tgp algorithm finds the predictive distribution for new sample points conditional on the points that have already been generated and evaluated by the search pattern. If the point recommended by tgp is a better point than those in the pattern, the search pattern continues from there; otherwise, it is discarded. When applied to our test problems, APPSPACK-tgp not only detected the global minimum, but was also able to generate a response surface that captured most second-order structure.

Significance

Our key third-year accomplishments were the:

1. Development of a multifidelity GP (GP-MF) model
2. Development of three new OUU algorithms
3. Successful demonstration of these GP and OUU algorithms on multifidelity test problems.

These algorithmic developments and implementations within DAKOTA will help the science and technology (S&T) community substantially reduce the computational cost of modeling, simulation, and design optimization efforts at Sandia and beyond.

In addition, our research into numerical methods has furthered our thinking about what is possible in different Sandia programs. For instance, the cost savings associated with the multifidelity GP models will allow us to fully explore the quantification of uncertainty in software packages (within Sandia's Verification and Validation Program and Sandia's S&T portfolio) that had thus far remained unanswered due to computational constraints. By extending the expected improvement and efficient global optimization within our OUU algorithms, we can now begin to address resource allocation, decision support, and margin quantification problems that were intractable until now.

Other Communications

L.P. Swiler, T.G. Trucano, W.L. Oberkampf, M. Pilch, K.J. Dowding, A.A. Giunta, and V.J. Romero, "Methodology Status and Needs: Verification, Validation, and Uncertainty Quantification," presented at the SAMSI Kickoff Workshop, Research Triangle Park, NC, September 2006.

M. Taddy, H. Lee, G. Gray, and M. Martinez-Canales, "Optimization with a Gaussian Process Oracle," presented at the SAMSI Kickoff Workshop, Research Triangle Park, NC, September 2006.

L.P. Swiler, "Gaussian Process in Response Surface Modeling," presented at the Society of Experimental Mechanics IMAC Conference, St. Louis, MO, January 2006.

G. Gray, "Verification, Validation, and Calibration of an Electrical Circuit Simulator," presented at the SAMSI Kickoff Workshop, Research Triangle Park, NC, September 2006.

B.M. Rutherford, L.P. Swiler, T.L. Paez, and A. Urbina, "Response Surface (Meta-Model) Methods and Applications," presented at the Society of Experimental Mechanics IMAC Conference, St. Louis, MO, January 2006.

G. Gray, "Derivative-Free Methods for Simulation-Based Optimization," presented at the NC State Mathematics Colloquium, Raleigh, NC, September 2006.

A.A. Giunta, M.S. Eldred, and L.P. Swiler, "The DAKOTA Toolkit and its use in Computational Experiments," presented at the NSF - SAMSI Summer School on Computer Experiments, Vancouver, Canada, August 2006.

Topology Optimization for Improving Sensor Performance

67021

K. R. Long, E. B. Cummings, P. T. Boggs, B. G. van Bloemen Waanders

Project Purpose

High-sensitivity microfluidic sensors and analyzers must have low sample dispersion, i.e., a slug of sample must remain concentrated as it flows through the device. A promising way to achieve high sensitivity is to design the shape of the flow channel to minimize sample dispersion. However, in many cases – such as that of an expanding channel – it is found that varying the shapes of the channel walls in a topologically invariant way yields little improvement in dispersion, and it becomes necessary to allow "islands" to grow in the channel. Growing an island is a topological change, and the problem of finding the optimal geometry becomes one of topology optimization.

The goal of this project was to develop efficient, robust algorithms for topology optimization with nonlinear physics, and then deploy these algorithms in high-performance software and apply them to design problems in microfluidics and other application areas.

By its nature, topology optimization is a large-scale Boolean programming problem; however, in that native form it is an intractable problem. Much of the art in topology optimization is choosing a formulation in real variables that closely approximates a Boolean field without introducing spurious local minimizers. In many problems, there is a natural reparametrization that is simple and works well. In structural topology optimization, for example, if the relationship between material density and stiffness is taken to be nonlinear and monotonic, then for certain objective functions there will be no payoff to intermediate material densities, so that the solution is naturally driven to a Boolean field.

In microfluidics and other problem areas with nonlinear physics, no such lucky accident happens, and we must deal directly with controlling the design variables so that they approach Boolean values. The obvious way to do this is with a penalization on any nonBoolean values, but that is easily seen to be nonconvex, and indeed, in practice it is found that such a penalization introduces numerous artificial local minimizers. Much of the work of this project has been to develop robust, efficient schemes for controlling the design parameters to produce a nearly Boolean design without being trapped in local minimizers.

Problems with large numbers – thousands to millions or more – of design variables require specialized algorithms and software for their solution. This project builds upon an earlier LDRD project on large-scale partial differential equation-constrained optimization in which development of such software was begun; further work on both algorithms and software was needed in the course of this project to adapt those ideas to the needs of topology optimization and to deploy them on production problems.

During the course of the project, we encountered a class of problems where for sufficiently narrow channels our optimal designs could not be manufactured. Imposing a manufacturability requirement through a constraint on the shape is not practical, so we dealt with this by incorporating the manufacturing process (wet etching) into the optimization loop. Optimized in this way, our designs were certain to be manufacturable.

FY 2006 Accomplishments

Improved Level Set Formulations:

In level set methods, the domain boundary is taken to be the zero level set of a field variable; this level set can freely undergo topological changes, making it a suitable descriptor of a domain in topology optimization. The critical issue for maintaining an approximately Boolean material permeability is to control the bandwidth of the transition region around the level set.

In his 2004 Carnegie Mellon University thesis [1], Cunha penalized the slope of the level set function; however, in the context of microfluidics we found that Cunha's method was unable to provide a sharp enough boundary. To solve this problem, we devised an improved formulation in which the bandwidth is controlled by an inequality constraint on the level set function's slope. Numerical experiments have found this to provide more satisfactory boundary resolution.

Adaptive limited memory algorithm:
With the level-set formulations described above, improved design variables can be found with optimization algorithms such as limited memory BFGS, a quasiNewton algorithm in which several previous steps are stored. The choice of how many steps to store is critical for performance: store too few, and the convergence rate is suboptimal; store too many, and the procedure is "locked" into a poor approximation by the presence of outdated information. We developed an adaptive strategy for choosing memory size that can reduce significantly the number of iterations required to converge.

Tailored Global Optimization:
While our improved level set formulation greatly reduces the number of artificial local minimizers induced by our smooth approximation to a Boolean field, the formulation is still nonconvex, and the physical problem itself has local minimizers. It is important to address global optimization; however, a brute-force search of such a large design space is impractical. We therefore developed search strategies tailored to shape optimization in which the search is done on a frequency-filtered design subspace, guaranteeing that each search direction yield a macroscopically meaningful change in device geometry. These search directions are then used in a hybrid tunneling and annealing scheme.

Software:
Shape optimization subject to constraints that are partial differential equations requires features not often found in large-scale simulation codes. Rather than make ad hoc modifications to each simulator needed in our problem, we developed a new type of simulation-building toolkit that is ready for optimization from the beginning. This package, called Sundance, allows rapid development of optimization-ready multiphysics simulators that are found to have runtime performance at least as good as existing domain-specific simulators.

Minimum-Dispersion Devices:
We produced designs for minimum-dispersion microfluidic expanders and displacers.

Etch-Compensated Devices:
The small angles encountered in a minimum-dispersion displacer cannot be manufactured using an isotropic wet-etch process. To compensate for this, we developed a design procedure in which the shape of the etch mask is the design variable, and the fabrication process is incorporated in the optimization loop. In this way, we were able to devise a design giving optimal performance after distortion of the design by fabrication effects.

Significance

This project has significance beyond the design of several minimum-dispersion devices. The approach to topology optimization developed for this problem is broadly applicable in engineering design as well as in areas such as image reconstruction. The adaptive limited memory BFGS algorithm can be applied to essentially any large-scale optimization problem, and has already been used (together with Sundance) in problems of radiation source location.

The result of broadest significance to come from this project has been the development and refinement of Sundance. In addition to its application to microfluidic topology optimization for this project, it has been used on a wide range of problems at Sandia and externally, including:

- Simulation-based optimal design of a pyroelectric demodulator (Sandia)
- Inversion-based location of a radiation source (Sandia)
- Inversion-based location of a chemical or biological toxin in airflow (Sandia)
- Research on medical imaging and flow control (Carnegie Mellon)

- Research on optimal control and parameter estimation for groundwater flow (North Carolina State University)
- Research on physics-based scalable preconditioners for Navier-Stokes flow (Sandia)
- Simulation and design of desalinization membranes (Sandia)
- Research on stochastic projection methods for uncertainty quantification (University of Southern California)
- Research on math-based software for self-optimizing finite element simulations (University of Chicago, Texas Tech)

[1] A. Cunha, “A Fully Eulerian Method for Shape Optimization with Application to Navier Stokes Flows,” PhD thesis, Computational Science and Engineering, Carnegie Mellon University, 2004.

Other Communications

K. Long, A. Skulan, S. Margolis, and P. Boggs, “Shape Optimization of Microfluidic Channels,” presented at the NECIS Seminar Series, Livermore, CA, June 2006.

K. Long, “Shape Optimization of Microfluidic Channels,” presented at Sandia’s Microfluidics Seminar, Livermore, CA, February 2006.

K. Long, “Symbolic Software Components for Rapid Development of High-Performance Simulators,” presented at the High-Performance Computing Seminar, University of Southern California, Los Angeles, CA, February 2006.

A Mathematical Framework for Multiscale Science and Engineering: The Variational Multiscale Method and Interscale Transfer Operators

79752

R. B. Lehoucq, J. C. Hill, M. L. Parks, G. Scovazzi, G. J. Wagner, A. Slepoy, P. B. Bochev

Project Purpose

An emerging consensus within the computational sciences is that simulation over a broad range of scales is needed for tomorrow's efforts in science and engineering. Multiscale mathematics is a systematic approach for analyzing the integration of heterogeneous models and data over a broad range of scales. We are conducting preliminary investigations of multiscale mathematics with impact to Sandia applications.

We originally outlined two goals: 1) development and analysis of an abstract mathematical formulation for multiscale problems and 2) evaluation and testing of this formulation on prototypical applications. We proposed a mathematical formalism founded on a discontinuous Galerkin (DG) generalization of the variational multiscale (VMS) method that addresses three important issues common to multiscale problems: scale representation, scale separation, and interscale communication. In particular, the concept of interscale transfer operators as generalizations of numerical fluxes at space-time interfaces is a focus.

We separate our accomplishments into two categories of multiscale problems. The first, continuum-continuum, includes problems that allow application of the same continuum model at all scales with the primary barrier to simulation being computing resources. The second, atomistic-continuum, represents applications where detailed physics at the atomistic or molecular level must be simulated to resolve the small scales, but the effect on and coupling to the continuum level is frequently unclear.

FY 2006 Accomplishments

We continued atomistic-to-continuum (AtC) coupling analysis for model problems (finite and zero temperature). This includes efforts to understand the effects of finite-size analysis (equivalently, the limit of the continuum theory), a mathematical taxonomy of AtC interfaces that employs the rigorous tools developed by the domain decomposition community. In particular, we began preliminary investigations for an AtC thermostat for modeling heat flow in molecular dynamics simulations. This involved the development of an algorithm that allows bidirectional coupling, and a software effort to allow simulations.

We also began preliminary investigations into coupling a peridynamic continuum theory with atomistic methods. The peridynamic theory of continuum mechanics allows damage, fracture, and long-range forces to be treated as natural components of deformation and so is naturally coupled with atomistic formulations. This reformulation is accomplished by replacing differentiation with integration within the equations of continuum theory. Finally, we investigated the role of uncertainty quantification, including probabilistic/stochastic information as a mechanism to insert fine-scale information.

We continued to explore two approaches for continuum-to-continuum (CtC) multiscale modeling that leverage the flexibility of DG methods with the VMS method: local VMS and hybrid continuous/DG methods. We also continued work in adjoint-based error estimation techniques for CtC coupling by formulating and implementing this approach within the DG/Sage framework and are in the process of exploring and evaluating this approach for canonical model problems. CtC methods were applied to wall-bounded turbulent flows with success and extensions to more complex flows were initiated. Finally, we are continuing work on mathematical analysis of the VMS-DG method.

Significance

Many important physical phenomena, such as deformation, failure, and turbulence are inherently multiscale processes that cannot always be modeled with traditional finite element analysis. Typically this inability is either because the continuum approximation acts on a broad range of scales, or because complex atomistic processes affect macroscopic behavior. In the former case, a more flexible finite element framework that allows a separation of fine-scale and coarse-scale representations can be employed. In the latter situations, one may consider an atomistic description to resolve the underlying physics.

Unfortunately, fully atomistic simulations of most domains of interest are computationally infeasible, so multiscale modeling methods coupling atomistic and continuum simulations are of interest. AtC coupling enables a continuum calculation to be performed over the majority of a domain of interest while limiting the more expensive atomistic simulation to a subset of the domain. Unfortunately, combining atomistic and continuum calculations is challenging because the former is based on individual nonlocal force interactions between atoms while continuum calculations deal with bulk properties of matter that represent the averaged behavior of a huge numbers of atoms. This requires methods to couple across length and time scales spanning many orders of magnitude from the atomic to the macroscopic.

Applications have been a driving force behind the development of AtC coupling methods. For example, an understanding of the failure of carbon nanotubes (CNTs) is needed in order to design CNT-reinforced composites. This has motivated AtC simulations of CNTs in order to model their physical properties and the effects of defects. In this case, a fully atomistic simulation is infeasible, so atomistic representations are used in localized regions where individual atom positions are important, and a less expensive continuum representation is used elsewhere. The two simulations are coupled through an interface or "handshake" region. Coupling of atomistic and continuum simulations provides a computationally efficient mechanism to investigate not only the behavior of crack tips at a fundamental level, but also other phenomena including grain boundaries and dislocations.

A more flexible finite element framework is a DG generalization of the VMS method that addresses the important issues of scale separation and interscale transfer operators. The latter is a generalization of numerical fluxes at space-time interfaces. Our work indicates that such a multiscale approach within turbulence simulation leads to more accurate results with fewer degrees-of-freedom compared to traditional subgrid-scale models. A related approach is a hybrid continuous-discontinuous Galerkin multiscale formulation. This will impact the ability to develop multiscale representations that can be directly used by second-order finite element and finite volume codes that are commonly used within DOE applications. Thus, the second aspect of our research is to develop new methods that either extend or extract multiscale representations from more traditional discretizations.

Refereed Communications

T. Hughes, G. Scovazzi, P. Bochev, and A. Buffa, "A Multiscale DG Method with the Computational Structure of a CG Method," *Computer Methods in Applied Mechanics and Engineering*, vol. 195, pp. 2761-2787, April 2006.

M. Parks and R. Lehoucq, "*Atomistic-to-Continuum Coupling*," SIAM News, Philadelphia, PA, September 2006.

S. Ramakrishnan and S. Collis, "Partition Selection in Multiscale Turbulence Modeling," to be published in *Physics of Fluids*.

Other Communications

G. Wagner, R. Jones, M. Parks, A. Slepoy, and R. Lehoucq, "Atomistic-to-Continuum Coupling for Heat Transfer in Solids," presented at the World Congress on Computational Mechanics, Los Angeles, CA, July 2006.

G. Wagner, "Atomistic-to-Continuum Coupling for Multiscale Solid Materials Simulation," presented at Mississippi State University, Starkville, MS, September 2006.

Microprocessor Extensions to Accelerate Scientific Applications

79753

K. D. Underwood, R. C. Murphy, A. F. Rodrigues

Project Purpose

This project focuses on designing and evaluating microarchitecture extensions to accelerate DOE scientific applications on traditional microprocessors.

Many assume that supercomputers will be built from microprocessors or vector processors. At best, the national laboratories hoped to influence processor parameters such as memory bandwidth, memory latency, and cache structure. In contrast, industry has demonstrated a willingness to support multimedia applications by adding multimedia extensions (e.g., Intel's SSE2). The continued growth in transistors per chip yields an opportunity to improve microprocessors by adding a "scientific computing" extension to the microarchitecture.

We are studying processor innovations that leverage the benefits of Moore's law to achieve higher sustained floating-point performance for scientific applications. The focus is on a scientific computing floating-point extension. An extension that improves sustained microprocessor performance will impact supercomputing by reducing the number of processors required to sustain a petaflop. Many are currently proposing 25,000 to 130,000 processors to reach a petaflop. Reducing the number required is critically important to many applications that do not scale to 25,000+ processors.

This project includes a collaboration with the University of Notre Dame. Notre Dame is exploring architectures that migrate computation into the cache structures, while Sandia is focusing on the microprocessor core. The results are being evaluated in both new technologies and traditional microprocessors.

FY 2006 Accomplishments

To further enhance the architecture, we took numerous measurements of architectural parameters. These include the state required for a group of instructions and the number of synchronization (communication) points between them. We also measured mechanisms for partitioning the graphs into smaller groups. Notre Dame applied some of these initial results in an innovation to move computations out to the processors cache hierarchy. Initial results have been promising.

We performed high-level simulations of alternatives for complex floating-point units to execute wide ALU (arithmetic logic unit) control instructions. These simulations have shown promise for the proposed techniques; however, further refinements are needed.

We began work on the integration of the proposed architectural features with conventional microarchitectures. One of the key technologies for integration with a microprocessor is the detection and integration of a new type of aggregating load instruction. We simulated the "intra-cache line gather" technique and found a remarkable 10-15 percent speedup. We are currently preparing a technical advance, as this is a remarkable speedup for a processor change.

Finally, we began work on integer acceleration techniques, since the integer unit can be a major bottleneck when a faster floating-point unit is added. The integer research considers a technique similar to wide ALU control instructions; however, analysis has indicated that a small number of dataflow graphs is sufficient to cover a large percentage of integer instructions. Thus, we are simulating a smaller number of more static configurations that are showing promise.

Significance

The fields of defense, energy, and science increasingly rely on simulation to reduce experimentation. Nationwide, simulation is stepping up to the challenge where extensive experimentation is impossible.

These simulations rely on high-performance supercomputers that are based on microprocessors. These microprocessors are known to sustain a low percentage of peak performance on scientific applications. This work seeks to improve the sustained performance of microprocessors on Sandia's applications.

Characteristics of CTH, LAMMPS (large-scale atomic/molecular massively parallel simulator), and ITS point toward an opportunity to dramatically enhance commodity microprocessors. This research is developing processor extensions to support scientific applications within DOE. Researching extensions for microprocessors will enable Sandia to provide leadership to the microprocessor industry. In turn, this will enhance the performance of microprocessors on DOE applications. Ultimately, the success of this research could yield a key enabling technology for reaching sustained petaflops scale computing. In addition, leadership in this area by DOE and Sandia may be harnessed by Defense Advanced Research Projects Agency efforts in architecture.

Refereed Communications

K. Rupnow, A.F. Rodrigues, K.D. Underwood, and K. Compton, "Scientific Applications vs. SPEC-FP: A Comparison of Program Behavior," in *Proceedings of the International Conference on Supercomputing*, June 2006, CD-ROM.

Data Mining on Attributed Relationship Graphs

79754
T. G. Kolda, A. S. Yoshimura, B. W. Bader

Project Purpose

Large-scale knowledge discovery problems in intelligence and threat assessment analysis have become increasingly challenging, and tools are needed to focus analysts' efforts on the most important and relevant data. The goal of this project is to provide novel tools for analyzing relationships in structured data, which we represent as attributed relationship graphs.

In recent years, a variety of powerful linear algebra techniques have been developed to analyze relationship information. For example, latent semantic analysis (LSA) uses a singular value decomposition to exploit the connections between keywords and documents to facilitate text retrieval and clustering. The focus of our work has been to extend these kinds of ideas in new ways, removing some of their limitations and increasing their power.

FY 2006 Accomplishments

Current algebraic methods for graph analysis are restricted to analyzing graphs containing a single node type or a single edge type, largely because these graphs translate to two-dimensional (2D, matrix) representations, which are amenable to linear algebra techniques. We extend these popular 2D techniques and capture metadata on the links by adding a third dimension (3D) to our data representation. Given this 3D representation of the data, we use multilinear algebra to decompose this "tensor" into a factored form.

A tensor decomposition can be thought of as a form of dimensionality reduction onto "concept spaces" or as a higher-order generalization of principal component analysis. The components help us to understand the overall structure of the data. Furthermore, the reduced representations can then be used as input to visualization tools, clustering methods, and so on.

This year we demonstrated our new approaches in several applications: web link analysis, social network analysis, and citation/author analysis. For web link analysis, we considered a graph of the web augmented with anchor text information. The analysis then revealed major topics of the web, making it useful for topic-sensitive query response by search engines and even automatic clustering of search results. In the social network analysis domain, we analyzed Enron email communications and grouped employees by business role and showed the communication patterns among those roles and their changes over time.

We also clustered the members of an Al Qaeda data set in a unique way based on a variety of disparate relationships among them. A similar technique was applied to citation data, which permitted various tasks, such as author disambiguation and journal prediction. Finally, we presented a radically new interpretation of LSA that allows for three or more classes of objects to be considered simultaneously (e.g., documents, keywords, and authors). The approach also allows for the inclusion of keyword-keyword or document-document relationships. With this capability, we can perform simultaneous text and link analysis or include more information in a recommender system.

Significance

This project has led to entirely new methods of analyzing complex data sets. There are a wide variety of applications in the many different Sandia mission areas that involve information analysis and decision support. Much of our work has been motivated by the needs of the intelligence community and we are working to integrate our methodologies into existing Sandia intelligence tools. Scientific data analysis is another area in which our methods will likely find use.

Refereed Communications

B.W. Bader and T.G. Kolda, "Algorithm 862: MATLAB Tensor Classes for Fast Algorithm Prototyping," to be published in *ACM Transactions on Mathematical Software*.

T.G. Kolda, B.W. Bader, and J.P. Kenny, "Higher-Order Web Link Analysis Using Multilinear Algebra," in *Proceedings of the 5th IEEE International Conference on Data Mining (ICDM05)*, pp. 242-249, November 2005.

T.G. Kolda and B.W. Bader, "The TOPHITS Model for Higher-Order Web Link Analysis," in *Proceedings of the Workshop on Link Analysis, Counterterrorism, and Security*, http://www.siam.org/meetings/sdm06/workproceed/index.html, April 2006.

B.A. Hendrickson, "Latent Semantic Analysis and Fiedler Retrieval," to be published in *Linear Algebra and Applications*.

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Other Communications

B.W. Bader, R. Harshman, and T.G. Kolda, "Temporal Analysis of Social Networks Using Three-Way DEDICOM," Sandia Report SAND2006-2161, Albuquerque, NM, April 2006.

D.M. Dunlavy, T.G. Kolda, and W.P. Kegelmeyer, "Multilinear Algebra for Analyzing Data with Multiple Linkages," Sandia Report SAND2006-2079, Livermore, CA, April 2006.

T.G. Kolda, "Multilinear Operators for Higher-Order Decompositions," Sandia Report SAND2006-2081, Livermore, CA, April 2006.

Multiphysics Coupling for Robust Simulation

79755

R. Hooper, M. M. Hopkins

Project Purpose

Sandia has developed a wide variety of simulation codes to support its critical missions. Examples include compressible flow (Premo), heat transfer (Calore), semiconductor device models (Charon), circuit device models (Xyce), solid mechanics (Adagio, Salinas), and incompressible and reacting flow (Goma, Aria, MPSalsa).

As designs become more reliant on simulation, a singular set of physics can no longer describe the system adequately, and codes must be coupled to achieve an accurate prediction. Difficulties abound due to multiple time- and length-scales and the variety of algorithmic choices when performing a coupling. Loose coupling strategies exist, starting with successive substitution (easy to implement), proceeding to intermediate coupling strategies including subcycling and Jacobian-free Newton-Krylov methods, up to the tightest coupling in a full Newton method that includes full interapplication sensitivities (typically requires a major code refactor). The success or failure of the predictive capability of coupled codes not only depends on the physical models, but also on the coupling strategy chosen.

The purpose of this project is to investigate the stability, accuracy, and efficiency of coupling algorithms and to develop new algorithms and toolsets to guide application developers.

FY 2006 Accomplishments

- We developed a Matlab interface to the NOX nonlinear library and its integration with the Sierra framework to create a rapid algorithm prototyping tool.
- We extended the prototype multiphysics test suite to include an interfacial-based coupling problem from the literature that demonstrates dramatic improvements using our coupling algorithms and provides for algorithm validation.
- We enabled NOX-based coupling within "Solution Control" in the Sierra framework using Aria-Aria coupling as a driver.
- We demonstrated the advantages of stronger coupling algorithms for a thermal actuator problem using Aria.
- We created a new coupled application, Premcal, in Sierra by coupling Premo and Calore to enable studies of conjugate heat transfer between compressible flow and the solid aeroshell.
- We presented our work at conferences, organized a minisymposium on coupling strategies, and interacted with external research groups at the forefront of this field.

Significance

This research includes fundamental studies that attempt to theoretically tie information requirements to the performance of coupling methods. We are performing basic research on both prototype equation-coupling systems (hyperbolic/elliptic/parabolic) as well as real-world problems in production application codes.

This research project could potentially impact many simulation initiatives at Sandia by producing more robust algorithms and more efficient coupling strategies over the current technology. Without the development of better coupling algorithms, Sandia may not be able to solve or accurately simulate systems of interest.

Refereed Communications

R.W. Hooper, C.C. Ober, and T.M. Smith, "Enabling Fluid-Structural Strong Thermal Coupling Within a Multiphysics Environment," in *Proceedings of the 44th AIAA Aerospace Sciences Meeting and Exhibit*, pp. 1-10, January 2006.

Other Communications

R.P. Pawlowski, R.W. Hooper, and M.M. Hopkins, "An Analysis of Multiphysics Coupling Techniques for Large-Scale Applications," presented at the AIChE Annual Meeting, Cincinnati, OH, November 2005.

R.W. Hooper, M.M. Hopkins, and R.P. Pawlowski, "Exploring Coupling Strategies Within a Multiphysics Environment Using NOX – An Object-Oriented Nonlinear Solver Library," presented at the 7th World Conference on Computational Mechanics, Los Angeles, CA, July 2006.

Simulation of Neutron Radiation Damage in Silicon Semiconductor Devices

79756

G. L. Hennigan, R. J. Hoekstra, P. T. Lin, J. N. Shadid, E. R. Keiter, J. P. Castro

Project Purpose

The effects of neutron irradiation on semiconductors, especially for short times after the pulse, are not quantifiably understood through experiment or current modeling. This project seeks to model full-fidelity stockpile devices in the Charon simulator, including critical neutron defect physics for both the semiconductor and dielectric materials in the devices.

Modeling the necessary drift-diffusion equations using stabilized finite element methods presents several critical hurdles. The exponential gradients of carrier flux and source terms in semiconductor physics require substantially more robust stabilization than flow problems. Also, extreme stiffness due to the wide range of timescales, from nanoseconds for the carriers (electrons and holes) to perhaps hours for defect species such as divacancies, requires highly robust, stiff time integration technology. Application of the massively parallel solver technology developed at Sandia will allow us to solve these problems of unprecedented fidelity and size.

We applied a stabilized Galerkin approach with some success to this problem; however, techniques such as mixed element methods and flux corrected transport (FCT) offer the possibility of greater efficiency and robustness. Multilevel preconditioning will be necessary for massively parallel scalability. This will be a critical area of research through application of algebraic multigrid and physics-based preconditioners. Finally, development of stiff time integration algorithms is necessary, possibly including techniques such as operator splitting.

FY 2006 Accomplishments

The primary focus of work this year has been on the modeling of realistic devices under neutron irradiation, primarily stockpile bipolar junction transistors (BJTs). Substantial research and development of algorithms has been accomplished as a part of this work.

- Full implementation of the capability for irradiation defect physics in the bulk silicon: we applied the complete set of known physical defects and properties (currently 34 species and hundreds of binary interactions) for realistic semiconductor devices, including the effects of dopant passivation and migration.
- We added and applied support for generalized sensitivity analysis through forward and reverse automatic differentiation to an irradiated diode problem, and we began work for optimization and automatic parameter extraction.
- We improved stabilization technology with the addition of discontinuity capturing, source lumping, and L2 (second order) projection algorithms leading to moderate improvement in accuracy for a given mesh resolution.
- We preformed mesh refinement studies to quantitatively define the accuracy and convergence of these algorithms.
- We improved automatic mesh refinement, including static load balancing of the refined mesh and improvement of the formulation allowing substantial improvement in scalability and solver convergence.
- Time integration was improved primarily through tuning of the second order method and tolerances as well as the addition of breakpointing techniques for discontinuities in transient problems.
- We performed transient simulation of bipolar junction transistors under pulsed neutron irradiation demonstrating short and long time-scale evolution of defects and their impact on device performance.
- We applied multilevel preconditioning through 2- and 3-level algebraic multigrid algorithms, leading to impressive scalability improvements for up to 1024 processors on the Thunderbird cluster and Red Storm.

- We added a "Pseudo-1D" capability and demonstrated it as a rapid, cheap tool for exploration of bipolar junction transistor physics and a lower fidelity model of the full irradiation physics problem.
- We coupled Charon to the Xyce circuit simulator, allowing fully coupled device-circuit simulation. This gives Charon the capability to include realistic boundary conditions on the device for comparison to experimental results. Additionally, this gives Xyce the capability to choose from two-dimensional, "pseudo-one-dimensional," and compact "spice style" models for the appropriate fidelity and performance of devices in an irradiated circuit, allowing multifidelity optimization of devices and circuits as well as verification of the lower fidelity models against the higher ones.

Significance

The primary impact of this work is on robust modeling of relevant electrical weapons components under gamma and neutron irradiation. Semiconductor device modeling plays a critical role in the success of the Qualification Alternatives to the Sandia Pulsed Reactor effort for weapons qualification in a reduced testing environment. The technology being developed under this project allows substantial improvement of modeling capability for this class of weapons components.

Additionally, there is currently no parallel scalable semiconductor device simulator available commercially. As the fidelity requirements increase for smaller devices substantially impacted by environmental effects such as radiation and temperature, very-large-scale simulation is necessary. The development of algorithms supporting massively parallel modeling of these devices at an unprecedented fidelity will have a major impact on future modeling both academically and commercially.

Refereed Communications

P.T. Lin, R. J. Hoekstra, G.L. Hennigan, J.P. Castro, and J. Shadid, "Massively Parallel Modeling of Semiconductor Devices in the Charon Semiconductor Device Simulator," presented at SIAM Parallel Processing, San Francisco, CA, February 2006.

Data Pipelining for Heterogeneous Data Fusion

79757

G. A. Gray, K. L. Sale, P. J. Williams, G. S. Davidson

Project Purpose

Significant advances in methods of data collection coupled with decreasing storage costs have motivated the collection of large volumes of data in areas such as genomics, proteomics, chemistry, and medicine. Extracting meaningful information from these data sets is a challenge that is often hampered by data types that provide different views of the same situation, by data sets that give complementary information despite appearing dissimilar, and by the wide variety of data collection and storage formats. Our project proposes the development and use of a data fusion technique called pipelining to meet these challenges.

Pipelining is a computational tool that manages the exchange of information among various data sources and applications. Its primary advantage is that it allows data to remain in heterogeneous databases and thus does not require disparate data types to be converted into one large database. This characteristic has prompted biotechnology companies to develop pipelining methods for high-throughput data analysis and has led to increased success in solving drug discovery and protein identification problems.

Our work focuses on using pipelining to automate the process of data fusion by extending the use of ensemble classifiers to disparate data sources. We developed an algorithmic framework that includes separate base classifiers for each data type and a variety of fusion methods for combining the individual classifications and acting as a global discriminator. Because our approach requires only the base classification results and not the specific underlying data, pipelining will be accomplished.

We are designing software to implement our algorithms and demonstrating its usefulness on a phosphorylation prediction problem. Meaningful phosphorylation prediction results would complement and contribute to the Microscale Immune Study Laboratory Grand Challenge LDRD project. In addition, our software framework could be applied to many of the informatics problems currently being studied at Sandia.

FY 2006 Accomplishments

We continued researching a set of techniques referred to as ensemble classification. Traditionally, such methods combine the predictions of different classifiers for the same data set. Our goal is to extend this work to combine the predictions of classifiers on disparate data sets. To meet the classification goals of our project, we designed and tested a theoretical framework. In our approach, one classifier is individually applied to a specific data set. Then, the results are fed to the fusion algorithm, and a global classification results. To test our approach, we considered the unweighted and weighted voting algorithms on a set of data from the University of California at Irvine Machine Learning data repository.

We concluded that just as for single data sets, ensemble classification can be beneficial in the classification of disparate data types. However, fusion techniques must be designed wisely to improve upon the results of the best base classifier. In particular, in weighted voting, the choice of weights must be carefully considered. Moreover, fusion techniques should be applied only after classification training is completed for each data type. These results were presented and well received at two different conferences.

To continue studying ensemble methods, we investigated the use of boosting for patient survival predictions using the statistical package R. We also studied island counting, an algorithm that assigns class predictions to a set of measurements while simultaneously discovering the measurement channels most responsible for partitioning the set into clusters.

We combined island counting and parallel-tempered genetic algorithms to produce a more robust code. We also defined an appropriate problem on which to demonstrate our fusion algorithm. The problem is related to protein phosphorylation, arguably the most important regulatory cellular event. Our goal is to better predict the sites of protein phosphorylation. We are assembling a set of algorithms that make prediction of phosphorylation sites for proteins and testing them using a data set from the Phospho.ELM database.

Significance

The overall goal of this project is to develop a generalized fusion algorithm that will allow efficient information discovery on large, multiattribute, disparate data sets. Such an algorithm has important applications to the environment, science, energy, and other areas that include complex data mining problems. For example, to answer questions about the environment, data is available regarding the climate, toxins in the soil, local human and animal populations, and so on. All these factors must be taken into account when making decisions that will effect the environment.

Another area of significance is homeland security. For example, the need for bioagent detection systems has introduced new challenges in interpreting the data generated by sensors. Moreover, the ability to simultaneously consider related information, such as intelligence reports or epidemiological and environmental data, may prove pivotal in confirming threats or identifying false alarms. A large-scale data fusion approach is needed to tackle such issues.

Finally this project is helping to advance Sandia's capabilities in informatics.

Other Communications

G.A. Gray, D. Gay, P. Williams, and K. Sale, "Ensemble Classification of Disparate Data Sets," presented at the INFORMS Annual Meeting, San Francisco, CA, November 2005.

G.A. Gray, P. Williams, and K. Sale, "Enhancing Information Extraction by Applying Ensemble Classification to Disparate Data Sets," presented at The First Annual Conference on Quantitative Methods & Statistical Applications in Defense and National Security, Santa Monica, CA, February 2006.

Emergent Distributed Tracking and Identification from Features in Wireless Sensor Networks

79759
T. H. Ko, M. C. Chen, R. C. Armstrong, N. M. Berry, J. J. Carlson

Project Purpose

The purpose of this project is to develop a distributed tracking and identification system designed for low-power wireless sensor networks (WSNs) and an appropriate hardware platform. As the demands of securing our nation's resources increase, WSN technology is increasingly called on to provide a more robust, informative, and cost-effective solution than previously available. We propose to extend the capabilities of some of the more advanced architectures being investigated involving the integration of image sensors in WSNs and in-network processing to conserve bandwidth.

This research is meant to leverage the work done in the fields of computer vision and distributed sensor systems to provide cutting-edge research to both these fields. The information gained by having a network of information-rich image sensors will add the necessary robustness and resolution in space and time missing in current computer vision systems. In-network processing of image data will provide more relevant information to the user faster than the current centralized approach without greatly sacrificing the lifetime of the system. A single sensor node will not attempt to solve the entire problem; the task of identification will be distributed across the path of the event through different nodes, effectively gathering information and naturally tracking the event.

We propose using features extracted from images as the event representation, which is shared among nodes, enabling the sensor nodes to understand their physical relationship to one another. Overlapping clusters emerge within the sensor network based on shared information, adding robustness against limited node and communication failure within the network as well as controlling bandwidth use and directing the behavior of future nodes along the path of the event.

FY 2006 Accomplishments

- Simulation: We analyzed the link algorithm through simulation on Matlab and the Sandia-created Umbra simulation package. We tested its performance with varying coverage and density of sensor nodes.
- 3D calibration: We developed a novel three-dimensional (3D) calibration algorithm that converges quickly and with minimal computation.
- Hardware acquisition: We acquired the necessary radio boards for our sensor system and adjusted the DISCERN adapter board to connect the different modules.
- Module communication: We implemented a communication protocol between the different modules in the sensor node.
- Implementation on sensor node: We implemented the algorithm and its supporting system functionality to analyze the performance of the algorithm on real systems.
- DISA roadmap/working group: We participated in the development of the Defense Information Systems Agency roadmap and working group to outline future directions for this type of research.
- Intellectual property protection: We filed a patent application for our technology.

Significance

This work provides the foundation for a scalable surveillance system to protect our national assets, including federal facilities, national borders, and infrastructure. The research conducted in this project concerning information-based links and 3D calibration allows a varying number of surveillance cameras to be placed in a nondeterministic fashion. The algorithms developed will regulate communication and determine relative orientation and position.

A Numerical and Experimental Characterization of Decontaminating Water Distribution Networks

93503

B. G. van Bloemen Waanders, J. C. Hill, L. J. Frink, L. K. McGrath, B. W. Bader, S. J. Altman

Project Purpose

The goal of this project is to investigate the role of biofilms in aqueous systems by developing multiscale and multiphysics numerical algorithms, validated through an experimental effort. The first phase of the project will be devoted to understanding individual components of the multiscale problem, including the design of the laboratory experiments. The next phase will focus on the maturation of these modules and start investigating coupling methodologies. Chemical reaction will be added to the molecular code and a more accurate biofilm model will be developed. Coupling of a convection-diffusion-reaction (CDR) model will be considered, in addition to development of inhomogeneous Navier-Stokes model. Our experimental effort will focus on additional annular reactor experiments, atomic force measurements, and a mesoscale test loop. The close coordination of validation and model calibration through experimental results will also be undertaken.

FY 2006 Accomplishments

We made significant progress in the area of molecular modeling, micro- and mesoscale numerical modeling, and validation through optimization and laboratory experiments. Specifically we 1) applied density functional theory to characterize the structure and thermodynamics properties of a prototype biofilm, 2) developed a CDR model to predict chemical transport in a coupled bulk fluid phase and biofilm region, 3) started experimental measurement using an annular reactor, and 4) implemented reduced space sequential quadratic programming algorithms.

We developed a basic biofilm model using density functional theory that consists of a semipermeable layer for extracellular polymer substance, two macromolecules representing the in situ bacteria and a possible contaminant, and one chemical species. In anticipation of having to calibrate and modify the model with new characterization data, we designed an extensible communication layer for Sandia's molecular theory code, Tramonto, so that we can conveniently interface optimization algorithms and other analysis methods, in addition to providing the flexibility of extending to multiphysics.

We developed a three-dimensional finite element-based optimal control of convection-diffusion-reaction dynamics. This model was designed to predict the dynamics in the bulk fluid. We developed an algorithm to model different diffusion constants for the bulk fluid and the biofilm through Henry's law.

The microbial concentration was modeled with a convection-diffusion-reaction model with the rate law for microbial inactivation, which was incorporated as a sink term in the CDR equation. Our laboratory work focused on conducting experiments in annular reactors to investigate the integration of potential pathogens into biofilms and effectiveness of removing the biofilms through chlorination. Experiments have varied shear force, the initial concentrations of pathogens, and the amount of colony forming units in the biofilms.

Significance

We developed fundamental capabilities for the characterization of biofilms in water systems, both numerically and experimentally. Although these basic components have limited use for other Sandia mission areas, the use of large-scale optimization, multiscale physics, and validation methods can be applied to many technical areas within Sandia. Large-scale optimization algorithms will be used to calibrate and validate the numerical model against experimental results. Eventually, we will use the same algorithms in controling decontamination procedures.

Many spatial and potentially temporal scales exist that dominate the physics of this problem. Special numerical algorithms will have to be developed to upscale certain dynamical phenomena from one spatial scale to the next. Finally, we will be able to leverage the underlying characterization of biofilms to a number of important applications, such as the health and food industries.

Refereed Communications

F. Shang, J. Uber, B. van Bloemen Waanders, D. Bocelli, and R. Janke, "Real-Time Water Demand Estimation in Water Distribution Systems," in *Proceedings of the Water Distribution System Analysis Symposium*, p. 1, August 2006.

S. Altman, L. McGrath, and C. Souza, "Interaction of Introduced Biological Agents in Water Distribution Systems," in *Proceedings of the Water Distribution System Analysis Symposium*, p. 1, August 2006.

Other Communications

B. van Bloemen Waanders, L. Frink, J. Hill, B. Bader, and S. Altman, "Numerical Simulation and Experimental Validation of Biofilms During Contamination Events," presented at the Water Distribution System Analysis Symposium, Cincinnati, OH, August 2006.

Distributed Microreleases of Bioterror Pathogens: Threat Characterization and Epidemiology from Uncertain Patient Observables

93505
J. Ray, H. N. Najm, K. D. Devine, Y. Marzouk

Project Purpose

The aim of this work is to create robust inference strategies to characterize an unfolding bioterrorist attack based solely on scarce patient data collected over a 2- to 4-day observation period during the early epoch of the outbreak. We are assuming that the attack occurs as a release of an aerosolized pathogen formulation and is not captured on environmental sensors because of (a) its small size; (b) the coarseness of its formulation, which causes quick precipitation; and/or (c) the lack of instrumentation in the vicinity of the release location. In such a case, the first intimation of an attack will be the first diagnosed patient. The approach considers sources other than patient data to be instrumental in forming hypotheses that are then proved/disproved based on patient data.

A particular requirement in this project is that these inferences quantify the uncertainty inherent in them due to the incomplete nature of the observation process. Our approach is based on Bayesian inference and develops inferences as probability density functions that automatically quantify the uncertainty. Bayesian inference also requires the evaluation of large multidimensional integrals where the integrand is noisy. This requires sampling and adaptive refinement of the domain of integration. We expect 20-dimensional integrals, and parallel computing is a must.

The project is designed to progress on two parallel fronts: 1) the inverse problem that develops the means to form and evaluate various hypotheses based on a model of the disease/pathogen, and 2) the forward problem, which is a model of the disease as it progresses in a population. For noncontagious diseases, an incubation period distribution suffices for the model. For contagious diseases, where the number of people infected in the initial release is a fraction of the infected/incubating population, we require a model that captures disease spread. Since we target the stochastic early epoch of an outbreak, ordinary differential equation-based deterministic models are unsuitable. Further, since the model will be evaluated repeatedly within an inverse problem solution, speed is of essence.

We use a social-network-based approach to disease propagation, using data (the network) developed under the National Institutes of Health Models of Infectious Disease Agent Study. People "nodes" spread disease to others at "location" nodes, while simultaneously contaminating the location. Computational celerity is achieved in two ways: 1) by clustering snapshots of the time-dependent social network over fixed intervals, i.e., time-averaging, and 2) sampling the clustered network to arrive at a smaller one.

Algorithmically, the clustering, sampling, and disease propagation over a network is done by representing it as a hypergraph. We are modifying/exploiting partitioning algorithms in Sandia's Zoltan suite to achieve this aim. Challenges exist in identifying which graph characteristics have to be preserved while clustering and sampling and in designing sampling and clustering techniques that actually preserve these metrics.

This project exploits DOE's expertise in uncertainty quantification, sampling, and parallel computing to solve a problem of national relevance. Further, the disease modeling approach is a novel twist on multiscale systems modeling where macroscale properties emerge from the interaction of many micromodels.

FY 2006 Accomplishments

We commenced work on both the hypergraph disease propagation capability and the inverse problem.

- We constructed a disease model that incorporates both the variation of disease "load" and its time-dependent shedding rate. It is parameterized and general enough to accommodate the typical evolution of anthrax and smallpox in humans.
- We also constructed the social networks along which the disease spreads; these networks are time-dependent and based on TRANSIMS-based population movement (and consequently contacts). We used a simple two-step time-integration approach. We integrated these components into a serial disease propagation code that is currently undergoing validation. The simulation capability does not involve any sampling and as such will generate the "standard" results against which later sample-based simulations will be benchmarked.
- Approaches to sampling and clustering are at a preliminary phase. We have small sampled networks that are at the threshold of being tested with the disease propagation model. This is the first step of the try-and-test iterations that will identify the graph metrics to be preserved and the "correct" sampling techniques to be adopted.

On the inverse problems effort, we finished conducting a scoping exercise to determine what one might infer from scarce, partially observed data.

- We established that even for moderately sized smallpox outbreaks (1000 index cases), one may predict the size and time of the attack with less than 1 percent of the infected exhibiting symptoms.
- For anthrax, where a model for the dose-dependent incubation period exists, we were able to infer the size, time, and dose received based on four days of observations.
- For realistic attacks where the population will receive a spectrum of doses, we can robustly infer an average dose for the infected/incubating population.
- The length of the observation period seems to matter less than the fidelity with which the structure of the disease's evolution is captured. Thus it is preferable to have data collected at 6-hour intervals rather than on a daily basis – even if it means a shorter observation period.
- We applied this inference capability to field data from the 1979 Sverdlovsk anthrax outbreak (70 index cases) and compared it with the widely accepted analysis by Meselson [1]. The cause is suspected to be an accidental release from a Soviet military facility. The time of inception (April 2, 1979) was correctly identified by our model with less than four days of data; inferring the size took an observation period of eight days. However, it should be noted that 1) the outbreak was very small (80 infected people), 2) the data regarding patients was reconstructed 12 years later via grave-markers and interviews (hospital records had been "scrubbed"), and 3) the progression of the outbreak had been modified by public health responses (antibiotic treatment), causing it to linger for 42 days. Given these caveats, we were pleasantly surprised by what the inference process gleaned from the first eight days of the outbreak.

Significance

To the best of our knowledge, this is the third publicly accessible study that has attempted to infer the size and time of inception of a partially observed, single-focus outbreak involving weaponizable pathogens on the Centers for Disease Controls' Restricted Agents List (1997). It is certainly the only one where a dose was inferred. It must, however, be cautioned that such metrics have modest (conventional) epidemiological relevance since they often deal with endemic diseases; spreading rates and mechanisms are considered more important. However, these are crucial in a bioattack setting with uncommon pathogens where emergency medical responses have to be planned quickly.

The primary differentiator between our approach and current curve-fitting-based methods is the quantified uncertainty. However, to set it apart, one needs to exploit the uncertainty to further a purpose. We believe that the main use for uncertainty quantification should be in designing response plans, although that is outside the scope of this project.

We believe that the primary future users of this project are concerned with homeland security, though it is also relevant to asymmetric urban warfare. In both cases, the primary potential lies in designing sustainable response strategies that optimize (for speed) the allocation and delivery of medical resources. The current approach, a broad and rapid response to a detected attack, leaves open the possibility of distributed microattack feints; designing broad and rapid responses to multiple simultaneous attacks may be expensive (and possibly unnecessary). Alternatively, a measured response (based on the inferences) coupled with suitable hedging of the uncertainties can lead to smaller, but far nimbler response plans, thus reducing casualties.

[1] M. Meselson, J. Guillemin, M. Hugh-Jones, A. Langmuir, I. Popova, A. Shelokov, and O. Yampolskaya, "The Sverdlovsk Anthrax Outbreak of 1979," *Science,* vol. 266, pp. 1202-1208, November 1994.

Other Communications

J. Ray, Y.M. Marzouk, H.N. Najm, M. Kraus, and P. Fast, "A Bayesian Method for Characterizing Distributed Microreleases: I. The Single-Source Case for Noncontagious Diseases," Sandia Report SAND2006-1491, Albuquerque, NM, March 2006.

J, Ray, Y.M. Marzouk, H.N. Najm, M. Kraus and P. Fast, "Estimating Bioterror Attacks from Patient Data: A Bayesian Approach," to be published in the *2006 Proceedings of the American Statistical Association*.

Large-Scale Simulation for Human Behavior Modeling

93506
N. M. Berry

Project Purpose

Analytical needs have expanded beyond topics such as technology and nonproliferation and now have a core central human element. Analysts need systems that incorporate operational social structure, including patterns of influence and resistance, terrorist groups, criminal groups, and general social organization to produce a massively parallel simulation for a given local or global conflict. In this project, we are developing new analytical tools and simulations to facilitate the understanding of these human elements that are scalable to realistic problem spaces.

FY 2006 Accomplishments

Developed a System Concept Design
We published a design document detailing objectives, constraints, and system architecture design for the demonstrator concept.

Designed and Implemented a Cognitive Model for Use in HPC Environments
We conducted a detailed analysis of the project objectives with high-performance computing (HPC) personnel to determine how the code would be implemented in an HPC environment. This was necessary because one of the major goals of this project is to simulate realistic populations composed of tens of thousands of agents. We had planned to use SCREAM (Sandia cognitive runtime engine with active memory); however, SCREAM, as originally implemented, was deemed by HPC personnel to be too large for efficient use in a parallel simulation.

SCREAM requires the use of the Umbra framework, and Umbra is currently only configured to run on stand-alone platforms. As a result, we designed and implemented a streamlined cognitive model, or Cognitive Framework Lite," that does not require Umbra and was designed for efficient use in the HPC environment. Cognitive Framework Lite uses text analysis tools based on STANLEY (the Sandia Text ANalysis Extensible librarY), which automatically generates cognitive models from a given text corpus.

Developed Tools to Analyze Spatial-Temporal Patterns in Semantic Data Sources
As an interim product, we developed an analysis tool that allows analysts to examine data collected from media sources to detect and understand spatial-temporal patterns (e.g., diffusion of information, patterns of influence).

Developed Web Spiders for Automatic Data Collection
The final product of this project will rely on information collected automatically from the web in the form of large numbers of text sources. We designed and built web spiders to automatically collect text from online media sources. Data collected included location and time stamps used for geolocation and temporal filtering.

Developed Objectives for Parallelization of Algorithms for Spatial-Temporal Analysis of Semantic Sources
We produced a document detailing objectives for subsequent development of the spatial-temporal analysis tool.

Created an HPC Prototype of Seldon
This task was originally scheduled for late FY 2007 but was moved to FY 2006 as it was deemed to be a high-risk element of the project. Efforts to parallelize Seldon are under way, including integration with Cognitive Framework Lite.

Significance

We created a solid foundation for incorporating the cognitive models and the multiagent system framework and tools for analyzing the data we will be feeding into the completed system.

This work extends and enhances existing investments Sandia has made in the areas of text analysis, cognitive systems, and multiagent simulations. Specifically this project has provided a foundation upon which we can build applications in the following areas:

Department of Homeland Security
- Preventing acts of terror by watching the flow of information regarding critical facilities (e.g., ports, power plants)
- Planning and analyzing the effects of public information campaigns

Intelligence Community
- Identifying terrorist cells and how they form
- Measuring sympathy for terrorist groups

Department of Defense
- Combating Iraqi insurgency/solving the improvised explosive device problem
- Special Forces training
- Planning and managing strategic communications

DOE and NNSA
- Understanding flow of information
- Managing public perception of essential infrastructure, e.g., Waste Isolation Pilot Plant, Yucca Mountain
- Understanding evolving and spreading interests of foreign technology consumers

Refereed Communications

T. Ko, J. Basilico, N. Berry, C. Forsythe, "Using a Cognitive Model to Represent Information in a Social Simulation," in *Proceedings of the Cognitive Modeling and Agent-Based Social Simulation*, 2006, CD-ROM.

Other Communications

T. Ko, J. Basilico, N. Berry, and C. Forsythe, "Using a Cognitive Model to Represent Information in a Social Simulation," Sandia Report SAND 2006-2045C, Albuquerque, NM, 2006.

J. Basilico, M. Peters, and C. Forsythe, "Large Scale Simulation and Analysis for Human Interaction Modeling," Sandia Report SAND2005-7078, Albuquerque, NM, 2005.

Network Architecture Design for Next Generation Supercomputers

93507

K. D. Underwood, A. F. Rodrigues, M. J. Levenhagen, K. Pedretti, R. B. Brightwell, K. S. Hemmert

Project Purpose

While Red Storm should meet its design goals, fundamental limitations in the network interface and router designs pose system-level challenges that preclude reaching the goals for a next-generation network. Specifically, we seek to achieve bandwidths of over 30 GB/s, latencies of 500 ns, and message throughput of 15 million messages per second per direction at an uncorrected error rate of less than 1 in 10^{23} bits. Success will require careful design of every aspect of the network architecture and revolutionary advances in network interface architecture.

We are systematically exploring the issue of network interface architecture and the associated requirements for the router. Our primary focus is on supporting MPI (message passing interface) to provide the bandwidth, latency, and throughput desired for next-generation systems. We are creating both the hardware designs and the system-level simulation to model them. Leveraging the team's experience from Red Storm, we began by creating a validated simulation model of the system level performance seen on Red Storm.

Beginning with a model of Red Storm hardware, we are seeking to systematically evolve the combination of hardware and software architecture to meet the performance and reliability goals. We will perform design trade-off studies to determine where hardware resources must be dedicated to achieve the desired performance (e.g., better DMA (direct memory access) engines, large or small MPI acceleration unit, and so on.) To provide further insight, we will leverage cycle-accurate simulations and field programmable gate array-based emulation of the critical hardware components. This will help us to understand both the timing of these components and how they will integrate into the system.

FY 2006 Accomplishments

We accomplished all of our goals for the first year and made excellent progress on creating a simulation of the Red Storm infrastructure. We are beginning to work on the acceleration technologies that will be needed next year.

Our first objective for the year was to create a simulation of the Red Storm network interface controller (NIC). A high-fidelity router model was completed and used in experiments, and the NIC model was completed and passed numerous performance validation tests. The current NIC model is running the Sandia branch of the Cray Red Storm software with minimal modifications.

We are simultaneously working on techniques to accelerate message processing that will provide the foundation for next year's milestones. An architecture for a generalized microcoded matching engine was completed and the initial microcode is written. Early next year we will complete a hardware description of the microcoded unit. We also examined high-level architecture questions associated with transmit side optimizations.

Finally, we created a lightweight application programming interface for network interface research and ported MPICH-1.2.6 to it. This will allow us much greater flexibility to explore new network architectures.

Significance

High-performance computing has become a fundamental underpinning of the homeland security, defense, energy, and science missions of the DOE. The long-term physical security and economic prosperity of the nation are dependent on the capabilities of supercomputers. The only common fundamental component among all supercomputers is a high performance network. This project is creating network technologies to achieve the performance desired by supercomputers at the petaflop scale and beyond.

The end products of this research are expected to provide the foundation for a next-generation supercomputer. Much as Cray Inc. integrated Sandia-developed technologies in operating systems (Catamount) and network systems software (Portals), we expect that a supercomputer vendor will be able to integrate the advances from this project as part of the next generation of network interfaces.

Refereed Communications

A. Rodrigues, K. Wheeler, K. Underwood, and P. Kogge, "Fine-Grained Message Pipelining for Improved MPI Performance," in *Proceedings of the Cluster 2006*, September 2006, CD-ROM.

R. Brightwell, D. Doerfler, and K. Underwood, "A Preliminary Analysis of the InfiniPath and XD1 Network Interfaces," in *Proceedings of the Workshop on Communication Architectures for Clusters*, April 2006, CD-ROM.

K. Underwood, "Challenges and Issues in Benchmarking MPI," in *Proceedings of the European PVM/MPI Users' Group Meetings*, pp. 339-346, September 2006.

Quantum Computer Architecture, Software, and Applications

93508
E. P. DeBenedictis, B. R. Hamlet, B. W. Dodson, C. P. Tigges, A. Slepoy, E. E. May, A. Ganti, M. D. Peters, R. C. Schroeppel

Project Purpose

This project launches Sandia as a contributor in the field of quantum information science. Quantum information represents a new technology direction that shows promise for improvements in sensors, communications, and computing. Sandia's thrust is to build devices capable of processing quantum information and combine those devices into systems that could be used for end-user purposes in science, defense, and the commercial sector.

Our objective is to understand and create ways of applying quantum-information-capable physical devices to important problems, while other researchers work to develop these devices (including the team on LDRD Project 93522, "Developing Key Capabilities for Quantum Computing.")

Quantum computers have been proposed as engines for certain cryptanalytic problems, physical simulations, and as a performance booster for computers in general. In this project we seek to understand and extend ideas for the application of quantum computers to these areas and to understand the application of Sandia physical science technology to the construction of a quantum computer.

Quantum communication has been proposed primarily as an "antidote" for quantum computers' cryptanalytic capabilities, although there are other uses. Our goal is to understand and extend ideas in quantum communications and to understand the specific application of Sandia physical science technology to the construction of a quantum computer.

FY 2006 Accomplishments

- Quantum chemistry: We made a substantive contribution to an exponential speedup quantum computer algorithm in chemistry. Collaborating with a group from the University of California at Berkeley, we improved the quantum algorithms and specialized these algorithms to ion trap quantum systems now under development at Sandia. These algorithms have been acknowledged as a valid use of quantum computers outside the cryptoanalytic area.
- Quantum software: We developed a program for coding and manipulating quantum algorithms, including simulation.
- Quantum circuits: We developed code for realizing a general quantum operation as a sequence of elementary gates (the quantum equivalent of a compiler).
- Numerical algorithms: We conducted research on reducing the number of qubits required for factoring.
- Quantum entanglement: We conducted studies on the basis and use of quantum information and entanglement.
- Quantum communications: After conducting an internal review of quantum communications opportunities, we expanded the scope of the quantum effort at Sandia to include quantum communications.

Significance

Our accomplishments will establish Sandia as a participant in the quantum information and computing research community. Sandia's quantum capabilities (including physics, mathematics, and computer sciences) will benefit mission areas of science and national security.

Referred Communications

B. Dodson, "When the Left Hand and the Right Hand Cooperate without Informing Their Owner," http://arxiv.org/ftp/quant-ph/papers/0605/0605080.pdf, May 2006.

A. Aspuru-Guzik, E.P. DeBenedictis, A. Ganti, P.J. Love, R.P. Muller, and A. Slepoy, "Simulated Quantum Computation of Porphyrin Excited States," Squint 06 Workshop, Albuquerque, NM, February 2006.

Electronics and Photonics

Characterization and Application of Dielectrics with Controlled Leakage

67022
C. W. Dyck, S. D. Habermehl, J. M. Jungk, J. R. Webster, M. R. Shaneyfelt, J. P. Sullivan, J. R. Schwank, M. T. Dugger, T. A. Friedmann, D. J. Dickrell III, J. C. Banks

Project Purpose

Micromachined capacitive switches are ideally suited for applications such as low-loss microwave phase shifters and tunable filters for use in miniaturized synthetic aperture radar, tags, miniaturized transceivers for autonomous sensors, and miniaturized satellite systems. They exhibit superior performance over solid-state switches in terms of static power dissipation, linearity, and signal loss, and they have lower ohmic losses than micromachined ohmic contacting switches. Presently, these devices are limited by early failures because of build-up of surface and bulk charge during operation.

The purpose of this project is to investigate and mitigate dielectric charging. We propose to engineer and study novel dielectrics with controlled conductivity to dissipate charge. We are studying how the dielectric composition and defect concentration affect charge trapping in conventional plasma-enhanced chemical vapor deposition (PECVD) films and amorphous diamond (aD) variants. Based on this information, we are developing the desired dielectric and leakage properties that are necessary for high-performance capacitive switches. We are studying bulk and surface charging, quantifying the relative contributions of tribocharging and charging from high-electric fields, and investigating differences in static and dynamic switch behavior.

Our analysis techniques include current-voltage curve measurements, characterization using a NanoTest tribology platform, irradiation by high-energy neutral particle and charged beams, and novel microelectromechanical systems (MEMS) test structures. This information is correlated to switch performance and lifetime in order to select candidate films for high-capacitive switch lifetime. More recently, we have been evaluating low pressure chemical vapor deposition (LPCVD) dielectrics because they contain less trapped hydrogen and higher breakdown electric fields.

In parallel to our dielectric studies, we began developing capacitive switches with air-gap dielectrics as a near-term means of circumventing charging in capacitive switches. By substituting a capacitor dielectric with an air gap, the designer trades off a decrease in stored electric field energy with the detrimental effects of dielectric charging.

Traditional capacitive switches that use dielectrics such as nitrides are capable of achieving from 10:1 to 100:1 ratios in the upstate-to-downstate capacitance; however, practical air-gap capacitors are limited to ratios on the order of 3:1. Although this results in structures that function poorly as switches, they are useful in applications such as tunable filters where large capacitance ratios are not always necessary.

FY 2006 Accomplishments

We designed, fabricated, and characterized air gap capacitive switches in FY 2006. In order to maximize the upstate-to-downstate capacitance ratio, we took advantage of the planarizing effect of our sacrificial layer when it surrounds topography on the bottom transmission line layer. We fabricated a circuit that was designed to tune from 23.3 GHz to 17.0 GHz (simulated values). This is equivalent to an upstate capacitance of 166 fF, a downstate capacitance of 400 fF, and a capacitance ratio of 2.4.

The circuit consists of a fixed series inductance, a tunable shunt capacitor, and a fixed shunt inductance. The tunable capacitor consists of a supported plate that spans a transmission line and two shunt electrodes. By moving in- and out-of-plane, the shunt impedance between the transmission line and ground is modulated. We fabricated five versions of this circuit with distances between the dimple and the shunt electrode varying from 5 µm to 25 µm, in increments of 5 µm. All five designs were evaluated in the upstate and the downstate from 0 to 26.5 GHz.

Measurements showed that switches with dimple distances less than or equal to 15 µm did not function properly due to interference between the plate and the transmission line and shunt electrodes. Measurements of switches with dimple distances of 20 and 25 µm were almost all functional. The measured tuning states were 22.5 and 17.2 GHz, close to the design values.

We began evaluating LPCVD dielectrics for possible application to capacitive switches. LPCVD dielectrics exhibit superior charging properties relative to PECVD dielectrics; however, they are difficult to integrate because of high processing temperatures. We tested the feasibility of these films by depositing LPCVD Si-rich and Si_3N_4 over tungsten. The Si-rich films were incompatible with the tungsten electrodes, and peeling resulted; however, the Si_3N_4 films survived processing and show promise for future applications.

We evaluated PECVD SiON films using the NanoTest platform, Auger, and x-ray photoelectron spectroscopy (XPS). PECVD SiON appears to have in-plane chemical inhomogeneity at the micron length scale that may impact the reliability from device-to-device. The devices that do not fail immediately appear to follow a power-law time-dependent dielectric breakdown (TDDB) model, and extrapolation of dielectric breakdown indicates that other failure mechanisms will dominate switch lifetime.

Compositional mapping indicates that the plasma treatment changes the near-surface composition of the dielectric films. Films that were exposed to plasma and films that were not exposed to plasma both tended to behave similarly with respect to time-dependent dielectric breakdown, showing similar breakdown characteristics. Additionally, the data is fit with existing TDDB models using Fowler-Nordheim tunneling mechanisms. At higher processing temperatures, evidence of dielectric degradation due to the oxygen plasma is minimal; however, at the lower processing temperature, the plasma-treated films appear to break down more quickly.

Significance

The studies of PECVD dielectrics add to the general body of knowledge about these films. Although LPCVD films have been studied extensively, fundamental knowledge of PECVD dielectrics is limited. Any studies of charge trapping, breakdown, and composition are thus valuable from a scientific sense and a device perspective. In the latter case, detailed knowledge of the failure mechanisms of capacitive switches is not available.

The use of LPCVD films in capacitive switches has received little attention due to the process difficulties. Our results indicate that their use may be possible. This could provide films capable of withstanding higher electric fields for longer lifetimes.

Air gap switches are significant in radio frequency MEMS for use in tunable filters in applications including miniaturized synthetic aperture radar, tags, miniaturized transceivers for autonomous sensors, and miniaturized satellite systems. The capability to apply high-reliability tuning structures translates to increases in the reliability of tunable networks.

Nano-g Accelerometers Using Nanophotonic Motion Detection System

67023

U. Krishnamoorthy, B. E. Keeler, D. W. Carr, R. H. Olsson, A. A. Talin, G. R. Bogart, L. L. Hunter

Project Purpose

Deformable gratings have long been studied as efficient light valves, mostly for use in display and communication applications. These structures have traditionally relied upon first-order gratings with feature dimensions larger than the wavelength of incident light that can modulate the power between the zeroth and first diffraction orders as the gratings are moved in and out of plane. Static subwavelength zeroth-order gratings have also been studied analytically and experimentally in prior work.

We looked at a different type of diffractive structure that uses near-field optical effects that are very sensitive to the motion of the grating elements, and we considered lateral motion in subwavelength gratings with nanostructured beams. These designs result in optical resonant structures that can dramatically modulate the intensity of the reflected or transmitted zeroth orders, while moving only fractions of a wavelength in the plane of the grating. Hence, these devices add considerable flexibility to the design and use of grating optical modulators in display technologies, optical switching, and subangstrom-scale motion detection.

The goal of this project has been to fabricate and characterize the behavior of these gratings and implement them in an inertial sensing system capable of detecting accelerations smaller than 10 ng.

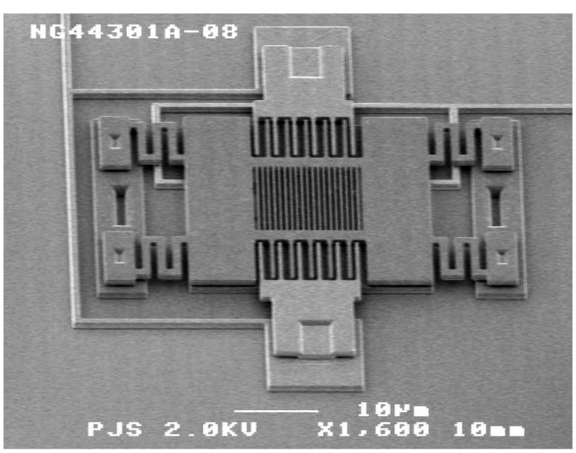
FY 2006 Accomplishments

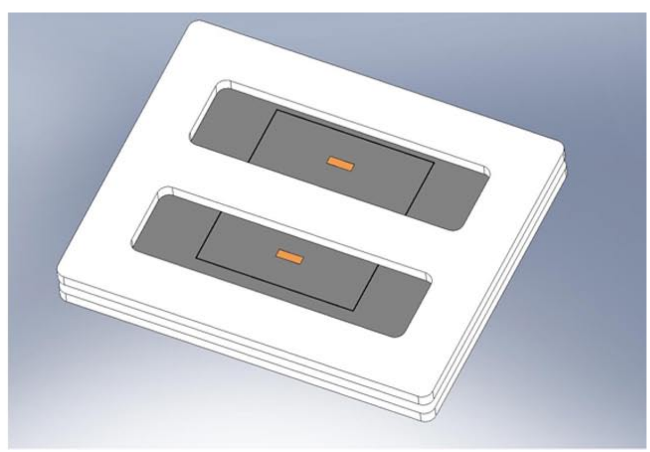
In the final year of this project, we developed several important processing capabilities, including:

- Depositing and patterning thick layers (20 to 40 microns) of extremely low-stress (as low as 15 MPa) epi-polysilicon on fragile Sandia ultraplanar, multilevel microelectromechanical systems (MEMS) technology (SUMMiT™)-based surfaced micromachined devices.
- Electroplating and patterning thick gold layers of extremely low stress (~ 20 MPa) on fragile SUMMIT-based surfaced micromachined devices.
- Directly integrating large substrate masses with nanograting sensors.

We successfully fabricated and packaged a high-sensitivity nanograting-based accelerometer. This involved several challenges:

- Optimizing DRIE tool for through wafer-etching of masses and springs





Optical nanophotonic sensor (Figure A) used to build a new class of in-plane, highly-sensitive (10 ng/√Hz), ultraminiature (< 1 cm^3) accelerometers in silicon (Figure B).

- Releasing accelerometers with fragile nano-gratings intact
- Packaging nanograting accelerometers to account for high sensitivity/impacts in handling.

We built nanograting-based accelerometers and verified operation of these devices on shaker tables with a laser doppler vibrometer-based system.

- We measured device resonant frequencies as low as 43 Hz, which corresponds to a thermal noise floor of ~ 10 ng/$Hz^{1/2}$.
- We directly verified acceleration measurements as low as 10 µg/$Hz^{1/2}$ for these devices and ran into operation limits of existing test equipment below these values.
- Previous nanograting sensor measurements pushed noise floors from 1.2 nm/$Hz^{1/2}$ to 50 fm/$Hz^{1/2}$ with custom-built circuitry. With this circuitry, this generation of integrated-mass nanograting accelerometers should be capable of measuring accelerations as low as 10 ng/$Hz^{1/2}$.
- We fabricated subwavelength gratings using nanoimprint lithography and used them for precise measurements of refractive indices.

Significance

Through this project we made several significant contributions to the science and technology community. First, we were the first to demonstrate femtometer-scale motion detection (12 fm/$Hz^{1/2}$) with a novel subwavelength optical sensing technique. We are also the first to build subwavelength grating-based accelerometers that can sense accelerations as small as 10 ng/$Hz^{1/2}$. No other devices or products come close to this level of sensitivity. The high sensitivity and small size of these devices enable a variety of applications in the areas of seismology and navigation. They also enable the design of compact 6-axis inertial measurement units (IMUs) with sensitivities comparable to the most sensitive IMUs built to date. This, once again, reinforces Sandia's place on the leading edge of scientific discovery.

Refereed Communications

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D.W. Carr, G.N. Nielson, G.R. Bogart, N.A. Hall, U. Krishnamoorthy, R.H. Olsson, B.E.N. Keeler, and J.J. Allen, "MEMS Inertial Sensors with Integrated Optical Transducers," presented at Photonics West, CA, January 2006.

Bragg Fiber Development

67024

J. G. Fleming, I. F. El-Kady, G. S. Subramania, S. S. Mani, T. S. Luk

Project Purpose

Guiding light through fiber has resulted in a billion-dollar fiber communications industry. There is a desire to integrate optical techniques with microfluidics for sensing applications important for fighting terrorism and national security needs. In order to develop a compact sensing package that can take advantage of a variety of optical and wet chemical techniques, optical signals produced by either laser-induced fluorescence or chemifluorescence need to be guided to a detector.

FY 2006 Accomplishments

We demonstrated the propagation and detection of an optical signal generated by the laser excitation of a fluorescent tag through the Bragg fiber. This accomplishment demonstrates the general principle proposed at the beginning of this project.

In order to confine and guide light to the detector, we developed (and placed on the wall) a hollow channel with a multilayer stack of reflective coatings. Bragg fiber, which can be fabricated on a silicon chip, has a narrow slit along the fiber as well as openings at the ends of the fiber that allow microfluidic integration from the top or from the ends. The volume inside the Bragg fiber may also be used as a microreactor.

We used laser-induced fluorescence and chemi-fluorescence to demonstrate the light-guiding functionality. For the laser-induced fluorescence experiment, fluorescence produced by laser excitation and guided by the Bragg fiber is detected by a spectrometer/photomultiplier system.

A 20 mM solution of Rh(VI) in dimethyl sulfoxide in the form of a liquid film is excited by a 532 nm laser at an intensity level of 3 W/cm^2. The film is made by sandwiching two drops of solution (0.0016 gm per drop) between a clean microscope slide and a microscope cover slip. The film covers an area of 18x18 mm. The thickness of the liquid film is calculated to be 11 µm. The fluorescence spot excited by the laser is visible when viewed through a laser goggle for 532 nm (orange color). Before measuring the transmission of the fluorescence, we measured the intrinsic loss of the fiber with the 532 nm laser and determined it to be 30 percent for a 1200 µm long fiber. Since the diameter of the fiber is about 25 µm, it is expected to be multimode.

In general, it is difficult to obtain good mode quality at the output of the fiber; however, with careful alignment, we can achieve a relatively clean mode. The fluorescence source is located about 200 µm away from the Bragg fiber end due to the thickness of the cover slip and a small air gap. The signal strength of the fluorescence viewed through the fiber is 100 times smaller than the laser excited fluorescence viewed directly. From this geometry, the solid angle accepted by the Bragg fiber is 6x10^{-3} steradians, whereas the solid angle sustained by the 20x microscope objective (NA = 0.4) is 0.6 steradians.

Significance

Through this work we established the theoretical background, fabrication processes, and performance needed to demonstrate a new, optical wave-guiding technology. The approach enables the propagation of light and liquid or gaseous species in a common platform. This is different from most approaches where light only evanescently couples into and out of the material of interest. In theory this should greatly improve the interaction between light and various liquids and gases. This could form the foundation for a sensor platform, high-Q spherical cavity for the investigations into the suppression of the local density of states, as well as novel optical structures where a relatively large propagation cross-sectional area is desired. However, the technology needs to be further optimized for each potential application.

Microwave to Millimeter-Wave Electrodynamic Response and RF Applications of Semiconductor Quantum Nanostructures

67025

M. Lee, C. Highstrete, E. A. Shaner, J. L. Reno, M. C. Wanke

Project Purpose

The purpose of this project is to characterize and understand the unique electrodynamic properties of quantum nanostructures in the frequency range of 0.1 to 100 GHz, which is the frequency range most relevant to modern high-speed computing, communications, and radar applications of high-speed electronics. The ultimate aim of the project is to develop sufficient fundamental knowledge to begin exploiting interesting electrodynamic response characteristics of nanostructures. This knowledge will enable us to prototype innovative radio frequency (RF) and microwave devices having radically superior performance resulting explicitly from quantum effects.

Specifically, we will work with nanometer-scale quantum wells, dots, and wires fabricated from high-mobility two-dimensional electron gases (2DEGs) in a III-V semiconductor heterostructure, as well as self-assembled low-dimensional nanomaterials such as one-dimensional carbon nanotubes. If pure enough and small enough, such structures can exhibit a range of purely quantum phenomena, including quantized conductance, Coulomb blockade, resonant tunneling, and Luttinger liquid behavior. These effects are marked by distinctive linear and nonlinear high-frequency conductance characteristics that can be used to detect and manipulate RF signals.

The peculiar advantage of quantum devices comes from the correlated, ballistic, and low-dimensional aspects of quantum charge transport. Devices based on these properties can approach ultimate physical limits on noise, responsivity, and bandwidth or speed. This project seeks to measure and understand the basic linear and nonlinear electrodynamic response of these semiconductor nanostructures, including the AC (alternating current) conductivity, noise properties, and nonlinear strength, about which surprisingly little is known.

Armed with this knowledge, we hope to begin designing wide bandwidth and frequency agile devices, such as fast direct detectors and heterodyne mixers, relevant to radar, remote sensing, and wireless communications technologies. The quantum nature of such nanostructures should allow these mixers to attain levels of sensitivity and gain-bandwidth not achievable with any standard semiclassical device.

This work leverages Sandia's strengths in microwave device characterization, growth of ultrahigh-mobility 2DEG heterostructures, and fabrication of nanoscale devices by both electron beam lithography and directed self-assembly. Several nanostructures have recently been fabricated with quantum transport signatures evident at temperatures up to 100 °K.

FY 2006 Accomplishments

We demonstrated that the resonant plasmon response observed in prior years in a semiconductor heterostructure quantum well can serve as an all-electronically tunable spectrometer-on-a-chip for far-infrared (FIR) frequencies near 1 THz. Excitation of resonant plasmon modes by FIR radiation in a quantum well transistor was used to analyze the spectral content of FIR illumination at frequencies between 0.58 to 0.99 THz.

A split grating gate design that allows localized pinch-off of the transistor channel greatly enhances FIR response over what has been previously demonstrated while maintaining the desired completely electrical tuning of the plasmon resonance. These advances enabled broadband FIR spectrum analysis without need for the mechanical motion of a mirror or grating. The parasitic resistance and reactance of the quantum well transistor were sufficiently small so that an electrical voltage ramp applied to the transistor gate could generate a spectrum at video rate speeds.

We fabricated sets of nanostructures for use as metamaterials at terahertz frequencies. These metamaterials are 2D arrays consisting of several thousand split-ring resonators (SRR) made of copper or gold on an undoped GaAs substrate. The SRR arrays are supposed to have negative index of refraction properties at certain resonant terahertz frequencies assuming the fabrication is of high enough quality. We developed SRR array metamaterials that have a better quality factor and more precise frequency match to design than have been made by anyone else. In addition, the construction of these metamaterials on GaAs allows photo-tuning of the metamaterial properties.

We made the first quantitative and broadband frequency measurements of the RF to microwave transmission and reflection coefficients of arrays of single wall carbon nanotubes. In doing so, we achieved signal-to-noise ratios and signal-to-systematic reproducibility ratios one to two orders of magnitude better than what is being reported in the scientific and engineering literature. Because of this very high signal and reproducibility, we were able to make the first detailed measurement of microwave loss characteristics in single-walled carbon nanotubes (SWCNTs).

Significance

Far-Infrared Quantum Well Spectrometer-on-a-Chip:
The FIR spectrum, roughly 0.1 to 10 THz, contains the resonance signatures of many molecules and materials. These signatures arise from quantized rotational modes (in chemical vapors) and lattice vibrations (in crystalline solids) that can be used to sense and identify a chemical or material with higher confidence than in any other range of the electromagnetic spectrum.

To sense FIR radiation power, many excellent detectors, principally bolometers and heterodyne mixers, are available. However, bolometers are not intrinsically frequency selective and hence require mechanical motion of external optics, such as mirrors and gratings, to generate spectral information. Mixers are frequency selective, but they cover only a narrow spectral range about a local oscillator (LO). This limited spectral range, and the fact that there are very few practical LO sources above roughly 0.5 THz, render mixers impractical for broadband spectral analysis.

The FIR analog of a compact, solid-state microwave or optical spectrum analyzer that continuously covers a broad frequency range with reasonable speed does not yet exist. Our demonstration of broadband and relatively fast spectral analysis using a completely electrically tuned new quantum device offers a potential solution to developing a practical, compact, reliable FIR spectrum analyzer.

Nanostructure Metamaterials for Terahertz:
Our successful fabrication of extremely high-quality SRR nanostructure arrays for terahertz metamaterials yielded the largest negative index of refraction ever achieved in the terahertz frequency range. In addition, the ability to turn the negative index resonance on or off by shining bursts of infrared light onto the metamaterial opens up the possibility of making a dynamically tunable terahertz filter or high-speed amplitude terahertz modulator using such nanostructured metamaterials.

First Measurement of Microwave Transmission and Reflection of SWCNTs:
High-purity SWCNTs may have unusual and conceivably very useful AC properties. Recently, microwave experiments studying rectification and conductance on transistors and diodes have been reported on devices formed from either individual or a small number of SWCNTs.

Interestingly, these works have generally concluded that either microwave loss is below measurement resolution or, if microwave dissipation exists at all, it is nearly frequency independent and hence is not the limiting factor in determining device bandwidth. Instead, empirically measured loss and bandwidth have been reported by others to be set by relatively large instrumental and device parasitic roll-offs and by systematic measurement variations.

Our new results show that SWCNTs do in fact dissipate microwave power in a frequency-dependent fashion. Our findings are the first to show quantitatively that SWCNTs are not “magic” lossless microwave materials, merely very good microwave materials.

Refereed Communications

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E.A. Shaner, A.D. Grine, M.C. Wanke, M. Lee, J.L. Reno, and S.J. Allen, “Far-Infrared Spectrum Analysis Using Plasmon Modes in a Quantum-Well Transistor,” *IEEE Photonics Technology Letters*, vol. 18, p. 1925, September 2006.

W.J. Padilla, M.T. Aronsson, C. Highstrete, M. Lee, A.J. Taylor, and R.D. Averitt, “Novel Electrically Resonant Terahertz Metamaterials,” to be published in *Physical Review Letters*.

C. Highstrete, E.A. Shaner, M. Lee, F.E. Jones, P.M. Dentinger, and A.A. Talin, “Microwave Dissipation in Arrays of Single-Wall Carbon Nanotubes,” to be published in *Applied Physics Letters*.

Development of GaN Power Amplifiers for SAR and Radar Fuze Applications

67026

A. G. Baca, M. E. Overberg, J. R. Wendt, M. Armendariz, R. J. Shul, P. F. Marsh, M. J. Rightley, C. E. Sandoval

Project Purpose

The purpose of this project is to develop power amplifier powers and power amplifier technology based on gallium nitride materials that will enable new capability in S-band (2-4 GHz) and Ku-band (12-18 GHz) radars.

FY 2006 Accomplishments

We demonstrated a 20 watt, S-band (near 3 GHz) power amplifier with 10 ns dynamic switching. We also developed technology for Ku-band power amplifiers, including developing and characterizing passive elements such as capacitors, resistors, and inductors. In addition, we developed circuit design models for these passive elements. And, finally, we developed through-substrate vias and generated transistor models for a 17 GHz design.

Significance

The significance of these accomplishments is two-fold. First, compelling performance of S-band (2-4 GHz) power amplifiers will enable more capable radars in the future. Second, the stage is set for achieving record-high power levels in Ku-band (12-18 GHz) radars.

Other Communications

A.G. Baca, C.E. Sandoval, M.E. Overberg, A.A. Allerman, S. Shinde, C.A. Sanchez, J.R. Wendt, P.F. Marsh, R.J. Shul, M.J. Martinez, M.G. Armendariz, G. Wouters, G. Kraus, R. Knudson, J. Lai, C. Rodenbeck, and C.T. Sullivan, “Stockpile Transformation with Gallium Nitride Power Amplifier Technology,” presented at NNSA Future Technologies Conference II, Washington, DC, October 2006.

Evanescent Wave Planar Photonic Biosensor

67027

D. W. Branch, I. Brener, M. J. Shaw, G. A. Vawter

Project Purpose

The overall goal of this project was to create a planar photonic biosensor as an analytical tool for rapid detection of a large number of biowarfare agents.

Given that the bioterrorism threat to national security has created an increased and immediate need for effective biosensors, a method to rapidly assess the presence and nature of a biological attack is crucial. In many cases the only way to unambiguously differentiate between a hostile attack and a naturally occurring appearance is through the forensic analysis of DNA sequences present in the suspect agent.

While the biotechnology exists today to perform this analysis, current methods rely on polymerase chain reaction or comparable target-amplification systems that require additional instrumentation and reagents, and more importantly, time, to carry out the procedure. A major challenge is the development of methods that do not require these sample preparation steps. Ideally a sensor would be able to detect a single strand of DNA containing the unique identifying base sequence, and further, would be capable of distinguishing between near-matches that differ by only one base pairing, i.e., single nucleotide polymorphisms.

Our goal was to combine state-of-the-art bioassay technologies with Sandia's own state-of-the-art photonics technologies to develop a compact, planar, arrayable, optically based sensor with unprecedented sensitivity. The sensor devices, fabricated in Sandia's Microelectronics Development Laboratory, will employ guided wave structures enhanced by the addition of ring-resonators. Our final goal was to determine the sensitivity of these devices for biological detection applications, as well as to characterize their behavior in fluids and environmental conditions.

FY 2006 Accomplishments

We successfully designed and fabricated a fourth-generation guided light wave device capable of interrogating fluid properties and operating as high-density immunoassay for detection of biowarfare agents. Through our work on low-loss, high-Δn, Si_2N_3/SiO_2 planar light wave circuits, we realized microring resonators with $Q > 2.4 \times 10^5$ fabricated in a standard CMOS (complementary metal oxide semiconductor) production facility. To that end, we created fourth-generation microresonator rings using the optimized design parameters to achieve stable and reproducible results.

Our new design uses identical pairs of microresonator rings, replicated four times in a vertical direction with varying waveguide-microresonator gaps ranging from 1.3 to 1.7 μm to improve resonator Q for operation in fluids. The design modification was essential to permit operation in fluids, given that the earlier designs had undesired fluid coupling as indicated by our measurements.

For routine detection in fluids, we incorporated two key packaging steps: a special microfluidic lid fabricated from polydimethylsiloxane using a glass mold and an on-edge fiber pigtail for die-to-die interrogation. Moving toward a more complete biosensor system, we also developed a readout scheme that used a laser servo-locked to the microrings' resonance to record wavelength shifts during detection.

Our initial experiments indicated that the environmental temperature variation perturbed the resonance sufficiently to cause drift in the resonance. We addressed this issue by using a temperature-controlled laser source and through temperature stabilization of the sensor element.

A critical aspect to our success was minimizing temperature instability through addition of a proportional-derivative thermal controller. We achieved a resolution of less than 0.0029 ° or 38.9 fm. This corresponds to a resolution of $\Delta n = 4.27 \times 10^{-8}$ for the index of refraction, allowing routine detection of less than 50 pg/cm^2 for each pair of microresonator rings. Our temperature stabilization approach improves over existing approaches and lends itself to field-portability. We successfully decreased the overall footprint of the system by using InGaAs photodiodes and a distributed feedback laser connected to the pigtailed microring resonators via fiber optics.

Moreover, we developed a method of calibration to characterize the temperature coefficient of the microresonator rings. We characterized NaCl solutions to assess system stability, flow sensitivity, and reproducibility. We detected bovine serum albumin protein concentrations ranging from 60 pg/cm^2 to 10 ng/cm^2. Moreover, the microresonator rings are sufficiently sensitive to distinguish between types of syringe pump used for introducing fluid.

Though temperature measurement was not a goal of this project, recent interest in microcalorimetry to study protein folding phenomena suggests that our devices would be an ideal analytical tool.

Significance

Our results indicate that planar photonic biosensors are exceptionally sensitive for fluid characterization and biological detection and have potential applications in protein folding and microcalorimetry. The relatively small sensing region makes these devices ideal for array applications, provided several packaging challenges are addressed.

Current photonic evanescent detectors rely heavily on fluorescent probes or prelabeled reagents to achieve detection and are limited to roughly four to eight elements. Our approach provides far more sensitivity while eliminating the need for fluorescent probes. The microresonator rings have several applications as analytical tools in the study of protein folding, where it is essential to detect minute thermal changes.

This work supports future biodetection systems to counter the production of biological weapons that threaten the peace and security of the US and its allies while establishing Sandia as the leader in biological detection.

Refereed Communications

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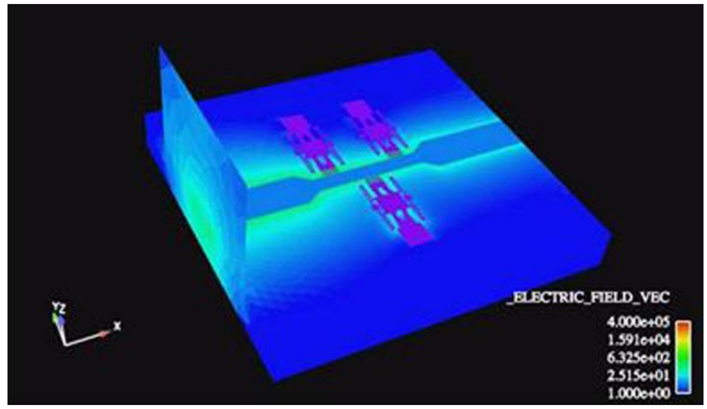
Passive Electronically Steerable Array for Miniature Synthetic Aperture Radar, Precision Guidance, and Intelligence/ Surveillance/Reconnaissance

67028

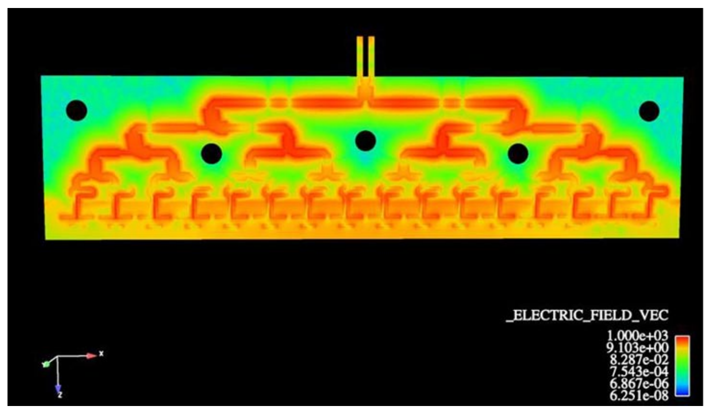
K. C. Branch, B. H. Strassner II, C. D. Nordquist, C. W. Dyck, G. M. Kraus

Project Purpose

The success of Sandia's synthetic aperture radar (SAR) systems has created greater demand for high-impact, sophisticated, multifunction radar systems. In parallel, radio frequency microelectromechanical systems (RF-MEMS) devices are maturing and have increased yield, reliability, and operation life. Realization and viability of both the components and microsystem applications require integration and miniaturization. Through this project we aim to solve these problems and stimulate breakthroughs in RF-MEMS and radar-phased arrays through the design and construction of a MEMS-based beam-forming network.



The electric field in the phase shifter device at one instant in time.



The electric field in the array elements and feed network at one instant in time.

Our approach was two-pronged, focusing on steerable antenna array design and component development and integration, both of which are geared toward next-generation applications. It is important that the component design be performed in conjunction with the system design. Phased arrays are versatile antenna systems due to their flexibility to manipulate magnitude and phase of the transmit and receive signal with amplifiers and phase shifters, respectively. However, integration of beam-steering components is not straightforward. Thus, construction of a passive, electronically steerable array (PESA) with RF-MEMS phase shifters is critical before an active array is attempted.

Ultimately, our motivation is to acquire all-weather, global positioning system (GPS)-denied day/night navigation and reconnaissance/surveillance capability with the flexibility of beam-steering and multiple operation modes (e.g., ground moving target indication). By integrating the antenna, feed-line, and beamformer, these advanced radar systems will be attainable in a timely and cost-effective manner with greater functionality and controllability. Novel capabilities such as antijam nulling, monopulse, multimode or multibeam operations require advanced array and beamforming architectures. Additionally, a compact and potentially conformal electronically steerable array (ESA) would negate the need for a heavy, mechanical gimbal, overcoming a major obstacle to achieving a 20-pound radar system.

FY 2006 Accomplishments

We focused our efforts on two primary areas in FY 2006: hermetic packaging of a 6-bit phase shifter and development of a lightweight antenna assembly.

Hermetic Packaging of a 6-Bit Phase Shifter
Based on our results from the previous year, we realized that a 4-bit phase shifter was insufficient for most applications. Thus, we created and electromagnetically modeled a new 6-bit phase shifter design. In addition, we realized that the reflect-line phase shifter architecture was too lossy and did not meet the desired wide-bandwidth performance.

- Our new 6-bit phase shifter design uses a true-time delay line architecture.
- We designed, fabricated, and partially characterized our new 6-bit phase shifter.
- Of the phase shifters that passed a minimal 4-of-64-state test, measured insertion losses ranged from approximately 1.5 to 2.5 dB, and the return loss ranges from approximately 8 to 12 dB.

  Without packaging, the phase shifters cannot be integrated into an antenna array and are exposed to air and water.
- We developed a custom packaging process that eliminates the lossy wire bonds we used to connect the phase shifters in our first prototype.
- We substituted two, packaged 6-bit phase shifters and a control sample for gross and fine leak hermeticity testing.
- Leak testing results showed bubbling in the two, packaged phase shifters from locations near the through-wafer vias.
- Because our vendor cut the via diameters too large, we were unable to fully cover the vias by metal pads during processing.
- The control sample passed both gross and fine leak testing, thus demonstrating the viability of the overall approach.

Lightweight Antenna Assembly
While the solid aluminum brick structure was a practical and simple approach, it was too heavy.

- We fabricated hollow plastic bricks using a stereolithography process and plated them with aluminum. The weight reduction was significant, yet the antenna performance remained virtually the same.

The brick structure was tedious to assemble and suffered from alignment issues.

- We designed a new assembly around the antenna cards that is simpler to build and does not suffer from alignment issues.
- The new array assembly is estimated to weigh less than 2 pounds and will fit on the miniSAR Twin Otter gimbal/platform.

Significance

This project will benefit RF-MEMS-based and SAR-based programs at Sandia and provide capabilities needed to meet Sandia's SAR roadmap and plans for a fully integrated radar electronics module-based SAR. The impact of this work will extend to benefit precision guidance, tags, communications, nanosatellites and other intelligence/surveillance/reconnaissance applications. In particular, successful completion of a PESA would prepare for immediate insertion into miniature SAR, ground moving target indicator radar, and tactical unmanned aerial vehicle systems on the 3-5 year horizon. This is possible due to the added functionality of an ESA in addition to reduced size, weight, and costs benefits. Additionally, the development of a phase-shifter package that is compatible with the antenna array assembly would enable future integration of Sandia's GaN power amplifiers in an active phased array system.

Refereed Communications

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Polymer Electronic Devices and Materials

67099

D. R. Wheeler, C. A. Apblett, S. M. Dirk, T. Wilson, B. L. Silva, V. Shulfer, G. R. Anderson, P. M. Baca, R. W. Olsen, F. P. Doty, K. Rahimian, T. M. Berg, J. D. Blaich

Project Purpose

The purpose of this project was to generate new methodologies for the fabrication of infrared (IR) photovoltaic devices.

Because about half of the sun's spectral power falls in the infrared range, conventional solar cells cannot collect the full amount of spectral power available. A method of increasing the utility of photovoltaics that has received much attention recently in the literature is incorporation of IR-absorbing lead sulfide (PbS) nanoparticles.

In 2002 photovoltaic devices accounted for between 427 MW and 560 MW of power worldwide. Devices based on crystalline silicon have reached efficiencies of 24 percent and, despite the high cost of production, silicon devices account for 99 percent of all photovoltaic devices. Hybrid cells, such as the Gratzel (photoelectrochemical), consist of hybrid inorganic semiconductors and a sensitizing dye molecule. An alternative to the silicon-based or silicon-hybrid device is an all organic-based device (all organic active area).

Typically, these devices work in the visible region of the spectrum. An improved method of making photovoltaic devices includes the IR region. We worked to address a number of issues associated with the fabrication of IR photovoltaics. We addressed the need to spin very thin coats of expensive and difficult-to-process conducting polymers. Current processing techniques often result in pinholes that short the device. We addressed the need to use dissimilar metal electrodes to generate internal electric fields to separate excitons in electron/hole pairs. We attempted to develop single-step routes to the formation of PbS nanoparticles and insoluble conducting polymers in a single processing step.

In simple photovoltaic devices, the primary exciton dissociation site is at the electrode interface. To improve on current devices, we modified the electrode surfaces in order to reduce these exciton dissociation sites and to improve efficiency. We focused on the functionalization of indium tin oxide (ITO), a transparent conductor that has played a crucial role in the construction of photovoltaic devices. Several reports showed successful modification of ITO using dipolar phosphonic acids, chlorosilanes, C60, 11,11,12,12-tetracyanonaphtho-2,6-quinodimethane (TNAP), tetraalkoxytin, and thiol-coated gold monolayers. We used functionalization with diazonium salts to modify the work function of ITO.

FY 2006 Accomplishments

- Developed the infrastructure to test IR solar cells
- Developed the instrumentation to look at electronic impedance spectra as a function of wavelength
- Duplicated work by Sargent et al. [1] as a starting point to building our IR photovoltaic devices
- Replaced one layer of the Sargent photovoltaic device with our bias-driven assembly of aryl rings. The layer we replaced was the thin layer of conducting polymer, which is prone to defects. Our process is a self-healing, defect-free process.
- Modified the work function of ITO using a variety of assembled diazonium layers. We moved the work function both up and down relative to native ITO. We generated layers with a 1 volt difference in work functions.
- Began documentation for publication and an invention disclosure
- Eliminated the need to use evaporated metal layers. We instead can now use ITO for both sides of the IR photovoltaic device.
- Built devices based on our new methodology. While not as efficient as Sargent's devices, the simplified fabrication has significant merit.
- Discovered that devices have a relatively short lifetime but have not yet identified the failure mode(s).

- Developed new synthetic methods to make PbS nanoparticles that will be amenable to other nanoparticles such as HgTe. This would allow the synthesis of HgTe without the use of the very toxic hydrogen telluride gas.
- Attempted to use both polymer precursor and nanoparticle precursors to thermally generate both the conducting polymer and the nanoparticles composite simultaneously from aqueous solution. While we made composites were made, problems with homogeneity require more time to address. Currently, the nanoparticles are soluble in toluene, and the conducting polymer is soluble in tetrahydrofuran (THF). THF made the nanoparticles precipitate and agglomerate. The conducting polymer is not very soluble in toluene. In situ fabrication of the composite matrix would be a major step to improved manufacture.

Significance

We demonstrated the ability to modify conducting and semiconducting surfaces using bias-driven assembly processes. We can modify the electronic behavior, specifically the work function of ITO. This work is critical to almost any hybrid organic inorganic electronic device, because it provides proof of the ability to potentially align band structures and manipulate electronic structure in a rational fashion.

We are exploring follow-on funding opportunities that will capitalize on the ability we have developed to control surface properties. We are also exploring opportunities to use in other areas the new nanoparticle synthetic routes we have developed.

The surface chemistry we developed has already found use in other programs. We are using the advances we made in this project to improve methods to functionalize electrode arrays for biosensing applications. We are going to examine the use of our chemistry to control interband surface states in silicon. The chemistry we developed will also impact nanoelectronic applications such as the functionalization of nanowires for sensor and electronic applications. We are also looking for space applications for some of our coating technologies.

This project has generated a great deal of new capability in the manipulation of interfaces. Interface control is critical to all new nanotechnologies. As devices shrink, the ratio of surfaces to volumes increases. Control of surface electronic behavior is, therefore, a very enabling technology for all nanotechnology applications.

Refereed Communications

[1] S.A. McDonald, G. Konstantatos, S. Zhang, P.W. Cyr, E.J.D. Klem, L. Levina, and E.H. Sargent, “Solution-Processed PbS Quantum Dot Infrared Photodetectors and Photovoltaics,” *Nature Materials*, vol. 4(2), pp. 138-142, February 2005.

Novel Photonic Crystal Cavities and Related Structures

79760

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Project Purpose

The purpose of our project was to develop a theory that would allow accurate modeling of the emission from thermally and electrically activated photonic crystals. The ability to modify various thermal emission processes is of critical importance to a number of energy and defense related projects. To facilitate the exploration of different photonic crystal topologies, we attempted to numerically calculate the density of photon states based on a real-space transfer matrix methodology that would allow the incorporation of dispersive materials such as metals.

FY 2006 Accomplishments

By combining an intricate quantum optics approach, electromagnetic transfer matrix approach, and numerical eigen-solution methodology, we developed a complete theory capable of predicting emission peak locations, relative heights, and temperature dependence. The results agreed remarkably well with experimental measurements. We also:

- Derived the analytics pertaining to this objective and incorporated them into a numerical code.
- Demonstrated the ability of the method in calculating the band structure for various two-dimensional photonic crystal structures.
- Derived the corresponding density of states and compared the results with experimental and available literature data.
- Worked out the analytics of extending the method to three-dimensional (3D) photonic crystal structures.
- Produced the corresponding 3D numerical code (still under testing).
- Incorporated 3D dielectric photonic crystal topologies.
- Extended the analysis to 3D metallic photonic crystal topologies.
- Evaluated stability criteria and code limitations.
- Cataloged the density of states for a variety of metallic photonics crystal topologies and the related crystal symmetry dependencies.
- Validated theoretical predictions using available experimental data on reflectance and transmittance of various fabricated structures.

Significance

The significance of our results is that we can model the thermal emission spectrum of photonic crystal structures, taking into account the absorption of the metal making up the structure. This is of critical importance in real-world applications and has already been applied to the modeling of thermal emission for application in a thermal photovoltaic project funded by the Defense Advanced Research Projects Agency.

This approach, initially developed for photonic crystals, should be applicable to the emerging field of metamaterials, where there is much work targeting structures active in the optical or visible regimes. However, little analysis exists as to how optical absorption by the metals used at these wavelengths will affect the performance of the devices being proposed. In fact, optical absorption is often simply ignored. It seems likely that we can further expand the tools we already developed into this field as well.

Refereed Communications

W.W. Chow, “Theory of Emission from an Active Photonic Lattice,” *Phys. Rev. A*, vol. 73, pp. 013821-9, January 2006.

W.W. Chow, “Active Photonic Lattices: Is Greater Than Blackbody Intensity Possible?,” presented at 36th Winter Colloquium on the Physics of Quantum Electronics, Snowbird, UT, January 2006.

Other Communications

W.W. Chow, “Microscopic Theory of Optical Response: From Thermal Emission of Photonic Crystals to Many-Body Effects in Semiconductor Lasers,” presented at Physics Department Colloquium, Texas A&M University, College Station, TX, April 2006.

Integrated NEMS and Optoelectronics for Sensor Applications

79761

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Project Purpose

The purpose of our project is to realize a compact sensor system, comprised of a microfabricated sensor, a vertical cavity surface emitting laser (VCSEL), and a photodiode. To accomplish this work, we will also seek solutions to the challenges presented by integrating micro- and nanoelectromechanical systems (MEMS/NEMS) with optoelectronics. By using microfabrication techniques in the realization of the MEMS/NEMS component, the VCSEL, and the photodiode, the system would be small and require less power than a macro-sized component.

In addition to the size reduction a microfabricated sensor provides, the microfabrication process can produce arrays of sensors that could be used to reduce noise in the measurement. An array would also provide redundancy and system robustness in inertial sensing applications. Therefore, the focus of this work is also to understand different methods of integration and how these methods address the physical challenges of placing optical components in near proximity to each other and integrating them with mechanically moving MEMS/NEMS components.

FY 2006 Accomplishments

The first milestone defined for FY 2006 has been satisfied, while the remaining milestones are at various stages of completion. We defined a process for the package-level assembly of the optical accelerometer system based on technologies that are extensible to a manufacturing environment. Specifically, we developed two different sensor designs that use SUMMiT V™ (Sandia ultraplanar, multilevel MEMS technology 5). VCSELs are commercially available, and custom VCSELs fabricated at Sandia, could be ported to commercial vendors for fabrication. In addition, the packaging techniques use commercial packaging technology, which should port directly to a commercial facility.

We are on track to achieving a measurement noise floor of 10 ng using board-level integration of the readout electronics. We fabricated devices with integrated masses. The new designed devices have temperature compensation in the supports of the devices, and we are beginning to test them as their fabrication and release is completed. We have initial measurement results with the device being excited by an impulse.

In order to integrate multielement photodiodes with the VCSEL on a single chip, we designed and fabricated a new VCSEL mask. We then tested the new design with a MEMS movable grating and found it to work well. We modeled and are fabricating new VCSEL designs to address the issue of feedback and system control in a macroscale system design.

We are developing a packaging scheme to integrate the electronics at the chip level and a process flow for integrating all pieces into a single encapsulated package.

Significance

Our work is being leveraged in two other LDRD projects that are developing new technologies for accelerometers and microphones. LDRD Project 67023 is developing a nano-g accelerometer using a nanophotonic motion detection system. Our packaging and detection scheme is being used to encapsulate the accelerometer and is furthering the testing capabilities beyond benchtop tests. LDRD Project 93518 is developing ultrasensitive directional microphone arrays. This application uses different optical component placement and a different detection scheme than the nano-g accelerometer. We are providing custom VCSELs and measurement techniques to accelerometers that are fabricated from the same design as the microphones but contain proof masses for acceleration transduction.

Development of Advanced UV Light Emitters and Biological Agent Detection Strategies

79762

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Project Purpose

Biological warfare agent detection with high sensitivity and low false-alarm rate is a critical capability for national security. The ideal bioagent detector is compact, robust, low maintenance, and supplies real-time feedback to enable its use in a wide range of situations. While fluorescence-based techniques are already powerful tools in biomaterials detection, such techniques are limited by the lack of compact, robust, efficient ultraviolet (UV) light sources at optimal wavelengths and the inability to effectively discriminate against background signatures in a compact system appropriate for field use.

The purpose of this project is to develop advanced solid-state UV light emitters and fluorescence-based bioagent sensor systems concepts that will address the performance limitations described above. We will apply our extensive experience in wide bandgap III-N materials to expand the range of light-emitting diode (LED) wavelengths down to 280 nm and shorter wavelengths, which offers higher fluorescence yields and enhanced discrimination for the detection of biological materials.

We will further develop deep UV laser diodes at deep UV wavelengths (~ 340 nm). We will implement these solid-state UV sources into an innovative fluorometer that will use modulation spectroscopy to measure lifetime-specific wavelength-resolved fluorescence spectra. This combined temporal and spectral resolution will enable effective discrimination of shorter-lived background signatures and specifically address the problem of high false-alarm rates that limits the effectiveness of the current generation of compact biosensor systems.

This is a high-risk, high-payoff effort that effectively integrates the world-class efforts at Sandia in nitride-based UV emitter development and UV fluorescence-based biosensor development to enable a new sensor capability of great relevance to national security.

FY 2006 Accomplishments

Our UV LED development efforts focused on identifying contributing factors to device degradation. Atomic force microscopy studies of the LED surfaces revealed that particular device structures had a nonplanar morphology that resulted in nonuniform current injection and early failure of the devices. Through modification of the p-layers of the LED structure, we were able to significantly improve surface morphologies and extend device lifetimes. LED structures incorporating these new p-type layers have demonstrated operational lifetimes of > 1500 hours at 30 mA. Output powers of 180 µW at 30 mA and with peak electroluminescence wavelengths of 270-280 nm have been demonstrated. We packaged three arrays of these latest generation LEDs for biosensor development efforts.

We initiated deep UV laser development efforts through the design and growth of AlGaN laser heterostructures on SiC substrates. We developed a substrate cleaving process for laser facets and built an optical pumping setup to evaluate the lasing properties of the materials. Our first optical pumping studies demonstrated stimulated emission at 338.8 nm from 1 mm long cavity structures. We also initiated AlGaN growth studies to develop effective p-type doping in Al = 0.30-0.60 alloys which will be required to achieve electrically injected 340 nm laser diodes.

Our biosensor effort included further development of our biosensor apparatus (fluorometer) and demonstration of biomaterial (e.g., tryptophan) fluorescence lifetime measurements using modulated UV LED excitation and phase-sensitive detection. We completed spectral characterization of biomaterial fluorescence using a compact spectrometer system.

This effort lays the groundwork for achieving simultaneous temporal and spectral resolution in our biofluorescence measurements.

Significance

The application of next-generation UV solid-state emitters to the detection of biosignatures will enable a broadly applicable, low-power, compact, low false-alarm-rate sensor that will address critical national security needs relevant to the Department of Homeland Security and to the Department of Defense.

Deep UV LEDs and laser diodes developed in this project can be used for water purification and surface decontamination as well as other applications of interest to national security. The nitride materials development strategies from this project are of interest to the general scientific community and can be applied to the improvement of other nitride-based devices such as solar-blind detectors.

Other Communications

A.A. Allerman, M.H. Crawford, S.R. Lee, D.M. Follstaedt, P.P. Provencio, K.H.A. Bogart, and A.J. Fischer, "MOCVD Growth of Al-Rich AlGaN Alloys: Materials for Deep-UV Emitters," presented at the 52nd American Vacuum Society Symposium, Boston, MA, November 2005.

A.A. Allerman, M.H. Crawford, S.R. Lee, D.M. Follstaedt, P.P. Provencio, and A.J. Fischer, "A Growth Method to Reduce Dislocation Density and Control Stress in AlN Films Grown on Sapphire," presented at the Materials Research Society 2005 Fall Meeting, Boston, MA, November 2005.

SMART Micropreconcentrator for Integrated Preconcentration and Detection of Chemical Agents and Explosives

79763

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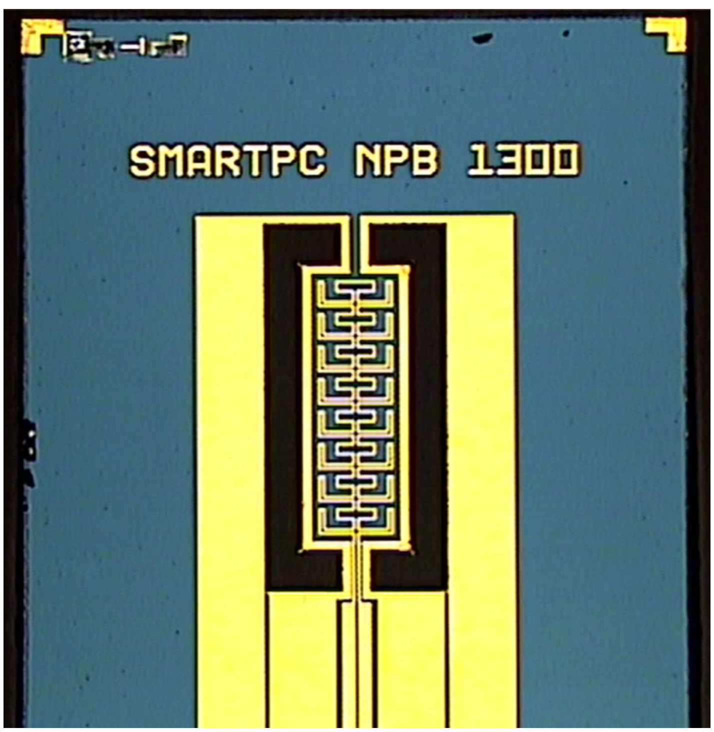
Project Purpose

The purpose of this project is to continue to develop a novel microdevice that serves as both a preconcentrator and a detector for microchemical analysis systems. The combination of a sensing and preconcentration function into a single device will enable microchemical analysis systems to perform more responsive analyses.

The SMART PC will be used as a front-end trigger that initiates sample analysis once it has determined that a sufficient sample has been collected. The Department of Defense desires diminished analysis times with increased ambient concentrations of chemical agents; current technologies either target the shortest collection time (highest concentration) or the lowest concentration (longest collection). By sensing collected mass, the SMART PC will allow variation of collection time to best suit the target ambient concentration.



A second generation uncoated SMART PC device that features large contact pads for easy fixturing and quick device preparation.

Using this technology, we shortened the analysis time of a simulant for a lethal concentration of Sarin to approximately 15 percent of the time required by a traditional microchemlab analysis. The device's mass sensitivity will also extend the dynamic range of microsensor systems by placing the desorbed analyte concentration in the linear region of detector response.

The SMART PC places a thin-film heater and adsorbent onto a Sandia-developed microelectromechanical systems (MEMS) resonator. The resonator consists of a small silicon plate compelled to pivot about torsion bars by Lorentz forces induced via a magnetic field. Mass collected in an adsorbent surface layer shifts the oscillation frequency. By placing a thin-film heater on this device, the collected sample can be thermally desorbed once the sensor indicates that an adequate mass has been collected.

Monolithic integration of the SMART PC, microgas chromatograph (microGC), and pivot plate resonator detector is on schedule to make the smallest gas-phase analysis system to date. The pivot plate resonator is similar to a SMART PC device, except that instead of a coating for preconcentration, it has a coating for sensing. Continued development of this microdevice may allow replacement of the surface acoustic wave (SAW) device in future microchemical analysis systems.

FY 2006 Accomplishments

- The new generation of devices we fabricated incorporates a better electrical connection scheme than earlier device generations, which will increase testing throughput and decrease fixturing cost and complexity.
- The device currently permits 11-second selective detection of dimethyl methyl phosphonate at the lethal concentration 50 (LC50) of Sarin. This detection is made with a SMART PC-GC-SAW system, which represents an 85 percent decrease in analysis time over a typical microchemlab system.

- Continued refinement of the sensing circuit has resulted in a factor-of-five decrease in signal noise.
- We performed an extensive study on different techniques and materials for device coating, including placing traditional sol-gels, nanoporous carbons, and DKAP polymers on the SMART PC surface, and testing them. Results from these experiments have yielded optimum coatings thicknesses and material performance.
- Hybrid integration of the SMART PC, microGC, and pivot plate resonator has progressed with fixturing developed for all of the components, and testing has begun on this assembled system.
- Monolithic integration of the SMART PC with a GC and pivot plate resonator has progressed, with successful fixturing and material coating of the sensing and preconcentration devices.

Significance

The SMART PC, as part of an integrated, low-cost platform, will help provide rapid and accurate identification of chemical and explosive threats for our military and homeland security agencies. Development of this device will enable microchemical analysis systems to operate more quickly and efficiently when exposed to deadly concentrations of target analytes.

The SMART PC is also an optimal, low-power sensor that can serve as a trigger for a remote and unattended system; the SMART PC's detection of a target analyte could cue a more advanced, dormant sensor system. In this capacity it would benefit programs in Sandia's nonproliferation investment area.

Finally, development of this unique resonator technology will provide a platform for future sensor efforts. Follow-on work leveraging our research is also being explored for National Aeronautics and Space Administration, Department of Defense, and DOE applications such as space sensing and stockpile stewardship.

Refereed Communications

P.R. Lewis, R.P. Manginell, D.R. Adkins, R.J. Kottenstette, D.R. Wheeler, S.S. Sokolowski, D.E. Trudell, J.E. Byrnes, M. Okandan, J.M. Bauer, R.G. Manley, and G.C. Frye-Mason, "Recent Advancements in the Gas-Phase MicroChemLab," *IEEE Sensors Journal*, vol. 6, pp. 784-795, June 2006.

Bead-Based Multiplexed, Orthogonal, Biowarfare/Infectious Disease Detection Microsystem and Technologies

84267

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Project Purpose

The purpose of this project is to create a WMD (weapons of mass destruction) detection microsystem, using novel components and a novel technology platform to more effectively solve the problem of rapid, sensitive, and specific detection of multiple target agents (chemical, biological, nuclear, and explosive (CBNE)) simultaneously in the same clinical or environmental sample.

While there are many detection system concepts being pursued (including others at Sandia) that can potentially, at least partially, address the issues of this problem, our technology platform and detection microsystem embodies unique advantages that should enable a more effective and complete problem solution. These advantages include: a bead with unique characteristics [a QD (quantum dot) bar-coded magnetic bead with surface functionalization to match the bar code], unique system components that use these characteristics for sample handling (mixing, sample cleanup), and two-stage optical detection (positive capture identification and bead bar-code reading) in a multichannel dielectrophoretic (DEP) alignment and bead-counting configuration.

FY 2006 Accomplishments

We made technical progress in three main areas:

Developed TTI (time to identify) analytical model of bead-based detection system performance to clarify design parameter variation effects and quantify system effectiveness.

- Applied model to three CONOPs (concept of operations)
- Detected pathogen (bacterial, viral, toxin) in 1 mL clinical sample
- Detected botulinum toxin in raw milk samples
- Detected airborne pathogen and explosives in aircraft cabin airspace
- Documented the model in a SAND report and a paper submitted to the *Proceedings of the National Academy of Sciences*

Developed surrogate chemical/biological reactions for WMD target detection using bead-based system.

- First set of reactions (direct assay) used streptavidin for the bead surface functionalization, with biotinated fluorescein as the surrogate target molecule. We achieved better than 0.01 femtomolar concentration detection using a confocal microscope detector.
- Second set of reactions (sandwich assay) used ovalbumin as the target molecule (representative of large toxin molecule), antiOva fluorescent dye (Atto-655) as the sandwich label, and streptavidin/biotinated antiOva for bead surface functionalization with similar sensitivity.

Designed, fabricated, assembled, and used (with surrogate chemical/biological reactions above) the first integrated micro/mesosystem versions of bead-based detector, including optical system for two-stage (external capture signature and internal bar code) and microfluidics system with gear pump, magnetic bead trap, and viewing area for optics.

Significance

We demonstrated that the key, high-risk, novel concepts that enable our system are feasible and practical. The system is meant to perform rapid, sensitive detection of multiple WMD targets in the same untreated sample.

Last year we demonstrated that QD bar-code magnetic beads could be synthesized. This year we demonstrated that beads could be surface-functionalized for two target molecules (direct assay and sandwich assay), and that the surface functionalizations could be detected using a two-stage optical detection mesosystem that operates as a small confocal microscope. We also demonstrated that the microfluidics required to pump a bead bearing liquid sample through the optical probe volume, where the two-stage optical detection occurs, can be built into a microsystem (less than 100 micron deep channels) that incorporates a magnetic trap for sample cleanup and a DEP focus section. The DEP focus aligns the beads at the correct cross-stream location and depth in the channel for optical detection.

The only remaining subsystem concept we haven't demonstrated is efficient mixing of magnetic beads for WMD target hybridization in the microfluidic format of our system. Concurrently with the key concept demonstrations, we have been building micro/mesosystems that integrate many of these concepts. So far we have integrated the optics (including software) and the microfluidics (without the mixer).

In addition, we haven't yet demonstrated robust operation of the entire system. This will be our goal for next year. Once the entire integrated system operation and all the key component operational concepts have been demonstrated, we will look to apply our microsystem technologies to problems in the area of WMD detection. Specifically, we are preparing to address two such problems with our bead-based platform: detection of botulinum toxin in raw milk (of interest to agricultural and homeland security), and detection of radionuclides (of interest to nonproliferation programs). In addition, we are pursuing funding opportunities for more broadly focused (multiple targets) threat CONOPs for explosive and biowarfare agent detection.

Other Communications

M.S. Derzon, "Status on Development of Platform for Clinical and Environmental Sample Acquisition and Multiplexed Agent CBNE Identification," in *Proceedings of the 2005 Scientific Conference on Chemical and Biological Defense Research*, pp.TS-3, 2005.

Terahertz Detectors for Long Wavelength Multispectral Imaging

84271

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Project Purpose

The main goal of this project is to advance the terahertz detector technology invented by Sandia and the University of California at Santa Barbara (UCSB) toward the goal of making a fast imaging array for multispectral imaging in the terahertz frequency range.

This detector concept is based on a unique grating-gated, high-mobility, quantum well field-effect transistor (QW FET) structure, where incident terahertz radiation excites resonant plasmon oscillations in the quantum well. The observed terahertz photoresponse is narrowband resonant with continuous electrical tunablity via an applied gate bias, and can therefore generate simultaneous illumination intensity and frequency content information.

We showed that the QW FET gate bias can be swept sufficiently fast that spectra across a 200 GHz range centered around 0.7 THz can be acquired with video-compatible refresh rates around 100 Hz. While we don't yet completely understand the physical mechanism responsible for the photoresponse, which hampers improvements in sensitivity and operating temperature, we have made significant advances. Our ongoing work focuses on elucidating the physical detection mechanism, raising the operation temperature above 77 K, enhancing the responsivity, and optimizing size and coupling to single pixel elements. In the final year, we will also begin integration into arrays for both multifunctional pixels and imaging.

Sandia is a world leader in growth and fabrication of these types of devices. Successful implementation of this technology into an array will enable both imaging of objects in the terahertz frequency spectrum and also determination of spectral properties. The ability to determine the spectrum of the terahertz radiation opens the possibility of distinguishing between materials being imaged as well as determining the temperature of the imaged object. Furthermore, the speed of these detectors enables fast image acquisition and tracking of moving objects, as well as communication capabilities.

FY 2006 Accomplishments

We made two significant advancements on the QW FET: increasing the responsivity of the detectors by a factor of 1000 using an innovative split-gate design, and demonstrating that the responsivity and reactance of the split-gate design enables the detectors to act as an electrically tunable terahertz "spectrometer-on-a-chip" that delivers spectra at video-rate speeds with no mechanically moving parts.

The original designs for the QW FETs suffered from very low responsivity to terahertz radiation. Thus a major milestone in FY 2006 was to explore ways to dramatically increase the responsivity while maintaining the tunability. The new split-gate QW FETs design builds on an observation we made about the device physics — the responsivity of the original QW FETs increased dramatically when the gate was biased near pinch-off, but tunability was lost. We then developed an innovative new split grating-gate designed to incorporate both electrical tunability offered by plasmon coupling and the enhanced responsivity seen near pinch-off. This increased the responsivity from a few mV/W in the original design up to a few V/W in the split-gate design, and maintained the continuous frequency tunability.

The terahertz analog of a solid-state microwave or optical spectrum analyzer does not yet exist. We made a major advance towards such a terahertz spectrometer by demonstrating that the QW FET has the sensitivity and speed characteristics to function as a "spectrometer-on-a-chip." The greatly increased responsivity of the split-gate QW FETs enables the QW FET resonance signal to be recorded directly on an oscilloscope. Of equal importance, the QW FETs have sufficiently small reactance that the detector can be run as a swept spectrometer with trace acquisition time compatible with video refresh rates of many tens of hertz with signal-to-noise > 10 dB, limited by the recording electronics.

Significance

There is growing interest in terahertz technology for a variety of commercial and national security applications. Spectrometer-on-a-chip capabilities offer the potential for much simpler and more compact spectrometers than currently exist. The demonstration of video-rate spectroscopy opens the possibility of multispectral imaging as well.

Improving the responsivity by a factor of 1000 was a significant achievement and brings the device closer to application use. We are already leveraging the speed of the detectors to perform proof-of-principle dynamic terahertz spectroscopy measurements for analysis of hypervelocity impacts.

Refereed Communications

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, "Tunable THz Detector Based on a Grating Gated Field-Effect Transistor," in *Proceedings of the SPIE Photonics West*, pp. 612006-1 - 612006-9, January 2006.

E.A. Shaner, A.D. Grine, M.C. Wanke, M. Lee, J.L. Reno, and S.J. Allen, "Far-Infrared Spectrum Analysis Using Plasmon Modes in a Quantum-Well Transistor," *IEEE Photonics Technology Letters*, vol. 18, pp. 1925-1927, September 2006.

Other Communications

E.A. Shaner, M.C. Wanke, M. Lee, A.D. Grine, and J.L. Reno, "Electronically Tunable Terahertz Detection Using Plasmons," presented at the American Physical Society March Meeting, Baltimore, MD, March 2006.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, J.L. Reno, and S.J. Allen, "Terahertz Detection Using High-Mobility GaAs-AlGaAs Heterostructures," presented at the Materials Research Society Spring Meeting, San Francisco, CA, April 2006.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, J.L. Reno, C. Highstrete, I. Brener, "Solid-State Microelectronics for the Terahertz Spectrum," presented at the American Institute of Engineering Conference on THz Systems III, San Diego, CA, November 2005.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, J.L. Reno, C. Highstrete, I. Brener, and S.J. Allen, "Terahertz Microelectronics: New Components and Applications," presented at Southeastern University Research Association Symposium on THz Applications, Washington, DC, June 2006.

M.C. Wanke, "Tunable Terahertz Detectors Based on Plasmons in Field-Effect Transistors," presented at R.G. Herb Material Physics Seminar, University of Wisconsin, Madison, WI, April 2006.

Advanced Optical Trigger Systems for Firing Sets in Nuclear Weapons

93417

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Project Purpose

The purpose of this project is to develop an advanced optically activated solid-state switch technology capable of surviving multiple cycles when switching loads of 5 kV/5 kA. The development of such a switch enables replacement of sunset technologies with a solid-state device that is less susceptible to shock and aging effects. Realization of this potential requires development of new optical sources and switches based on key Sandia photonic device technologies: vertical-cavity surface-emitting lasers (VCSELs) and photoconductive semiconductor switches (PCSSs). The purpose of this project is to develop new VCSEL light sources and new PCSS devices that together function as optically activated switches capable of operating for multiple shots under 5 kV/5 kA load conditions.

It was shown previously that the switching capacity of a PCSS device could be increased from 100 A to1 kA by distributing the current into multiple filaments triggered by an array of approximately 10 high-brightness, line-shaped illuminators. This historic demonstration was limited by the ability to manufacture arrays of mechanically-stacked edge-emitting lasers with sufficient optical uniformity.

In VCSEL arrays, adjacent lasers use identical semiconductor material and are lithographically patterned to the required aspect ratio. However, we demonstrated that good optical uniformity in rectangular-aperture (e.g., 5 by 500 µm) VCSELs is difficult to achieve due to the lack of optical confinement in the long dimension. We plan to apply effective index techniques, pioneered at Sandia for leaky-mode VCSELs, to create a lateral photonic lattice that selects a single transverse mode with high brightness and uniformity. These sources will be developed and tested with complementary PCSS designs employing interdigitated multifilament contacts for high-power switching.

FY 2006 Accomplishments

We focused on developing a better understanding of the illumination requirements for stable triggering of multiple-filament PCSS devices. We completed the fabrication and characterization of uncoupled VCSEL arrays, making low- and medium-density arrays of VCSELs with fill factors of 4 percent and 20 percent, respectively. We also successfully triggered a PCSS device with an array of uncoupled VCSELs, demonstrating the first successful triggering of a PCSS device with a VCSEL array. Finally, we have begun to quantitatively characterize PCSS triggering versus array fill factor, optical pulse energy, and with two or more parallel filaments.

The laser development effort was directed toward providing well-behaved VCSELs for use in initial PCSS triggering experiments. We successfully fabricated and characterized uncoupled linear arrays of VCSELs having optical fill factors between 2 percent and 20 percent. We mounted a single eight-element VCSEL array, with 4 percent optical fill factor, onto a high-voltage RF connector and drove it with a 310 V, 10 ns pulse from a 50 ohm source, yielding an optical pulse energy of 4 nJ incident on the PCSS device.

Significance

This solid-state switch technology will result in improved component manufacturability, reduced size, parts count, and enhanced surety of firing sets. It will advance the science and technology of compact high-brightness VCSEL arrays that will see application in systems for DOE and the Department of Defense. Finally, our understanding of semiconductor physics at high voltage/current levels will be improved.

Refereed Communications

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D.K. Serkland, G.M. Peake, K.M. Geib, R. Lutwak, R.M. Garvey, M. Varghese, and M. Mescher, “VCSELs for Atomic Clocks,” in *Proceedings of the Photonics West 2006*, no. 613208, January 2006.

D.M. Grasso, K.D. Choquette, D.K. Serkland, G.M. Peake, and K.M. Geib, “High Slope Efficiency Measured from a Composite-Resonator Vertical-Cavity Laser,” *IEEE Photonics Technology Letters*, vol. 18, pp. 1019-1021, May 2006.

P. Devgan, D.K. Serkland, G.A Keeler, K.M. Geib, and P. Kumar, “An Optoelectronic Oscillator Using an 850 nm VCSEL for Generating Low Jitter Optical Pulses,” *IEEE Photonics Technology Letters*, vol. 18, pp. 685-687, March 2006.

Robust Tunable Multifunction Amplifiers Using GaN and RF MEMS Technology

93510
C. D. Nordquist, G. M. Kraus, A. G. Baca

Project Purpose

Radio frequency systems that are adaptable to their environment hold potential for improved performance, greater efficiency, higher data security, and greater functionality. These systems can be used for multiband communication and radar systems for imaging, tagging, fuzing, and guidance. A key component in these systems is reconfigurable circuits such as power amplifiers, low-noise amplifiers, and mixers. Potential examples include amplifiers with low noise and power capabilities, tunable frequency and bandwidth, multiclass operation, and adaptability to environmental changes.

The purpose of this project is to develop an integrated technology for realizing the benefits of these reconfigurable circuits. The overall goal is to integrate active devices with low-loss tuning elements. While the active technology can be any high-speed electronics, GaN field effect transistors offer very high-power density and low-noise. Tuning can be achieved using many types of devices, but radio frequency microelectromechanical system (RF MEMS) devices offer best-in-class loss and linearity.

We will demonstrate a monolithic GaN amplifier using RF MEMS technology to switch between two modes: low noise and power. An amplifier with 0.6 mm gate periphery should have < 1.5 dB noise figure and > 2 W output power. Intermediate goals include a high-efficiency switched mode amplifier using GaAs transistors and RF MEMS, a hybrid switched-mode low-noise amplifier/power amplifier (LNA/PA) using GaN and RF MEMS, and fixed GaN LNA and PA monolithic microwave integrated circuits (MMICs). Most importantly, the integrated capability will be available for realizing reconfigurable microwave circuits for any application.

These circuits will demonstrate a unique capability in reconfigurable circuits, which will be a differentiating technology for Sandia. As a result, Sandia will be in a unique position to address the tunable circuit needs for future systems such as lightweight radars, secure communications, telemetry, and miniature tags for IFF (identification, friend or foe).

FY 2006 Accomplishments

We met the majority of our FY 2006 milestones. In partnership with the University of Colorado at Boulder, we demonstrated the first Class-A to Class-E switched-mode power amplifier using a commercial GaAs transistor and Sandia-fabricated RF MEMS matching networks. We characterized the output impedance of the GaAs transistor at Class-A matching conditions for best linearity and at Class-E matching conditions for best efficiency, designed and fabricated matching networks in the RF MEMS technology, and assembled and tested the amplifier against fixed amplifiers in each operating mode. The RF MEMS tuning generated the expected result, with minimal degradation of the amplifier due to the low loss of the switch.

To demonstrate a dual-mode GaN LNA/PA, we completed full noise and load-pull characterization of 0.6 mm gate periphery GaN transistors for output power at S-band. We also completed additional characterization of the GaN device's efficiency as a function of bias to allow for design of an amplifier using adaptive bias for optimum efficiency across a broad range of power levels.

Finally, to progress towards a monolithic reconfigurable amplifier, we compared the GaN MMIC process flow and the RF MEMS process flow and found that them compatible for fabricating GaN/RF MEMS-based adaptive MMICs with only two minor process changes. We designed the RF MEMS switches in the GaN MMIC process and expect to fabricate them in early 2007.

Significance

Reconfigurable GaAs power amplifiers represent one of the first applications of MEMS tuning to reconfigurable amplifier applications. Our accomplishments helped to define the trade-offs and potential associated with this particular technology. We received interest from commercial component suppliers in this reconfigurable amplifier technology due to the stringent demands of the handset and personal communication markets.

The characterization of the GaN transistors contributes immediately to internal Sandia efforts in power amplifiers for radar and communications. Understanding the device performance and efficiency as a function of bias will help to define future power amplifier designs and system requirements.

Refereed Communications

P. Bell, C. Dyck, and Z. Popovic, "MEMS-Switched Class-A-to-E Reconfigurable Switched Amplifier," in *Proceedings of the 2006 Radio and Wireless Symposium*, pp. 243-246, January 2006.

Other Communications

P. Bell, "Reconfigurable Power Amplifiers," University of Colorado Boulder, Boulder, CO, June 2006.

Bloch Oscillations in Two-Dimensional Nanostructure Arrays for High-Frequency Applications

93511

W. Pan, J. L. Reno, S. K. Lyo, R. G. Dunn, M. C. Wanke, M. Lee, J. R. Wendt

Project Purpose

The purpose of this project is to investigate the physics and device applications of Bloch oscillations (BO) of electrons engineered in high-mobility quantum wells patterned into a lateral periodic array of nanostructures, i.e., a two-dimensional (2D) quantum dot superlattice (QDSL). Depending on the direct current (DC) electric field and the superlattice period, the BO frequency can be continuously tuned from the millimeter wave frequency range to the lower terahertz regime. The ultimate goal is to enable solid-state, electrically biased, tunable, high-frequency sources, amplifiers, and detectors.

Physically, a BO occurs when an electron moves out of the Brillouin zone (BZ) in response to a DC electric field, passing back into the BZ on the opposite side. This results in quantum oscillations of the electron, i.e., a high-frequency alternating current (AC) in response to a DC voltage. While tantalizing indirect evidence of BO's has been obtained in a quantum well superlattice (QWSL) with complex optical techniques, to date, no direct electrical transport evidence has been observed.

The primary difficulties preventing BO in the old QWSL approach are fast damping of oscillations by electron-optical phonon scattering, and electric field domain formation. In QWSL's, optical phonon scattering can be suppressed by designing the minibands below the optical phonon energy. However, since the electron motion parallel to the layers is not quantized, there is no true upper band edge for the minibands, and highly inelastic optical phonon scattering can still take place even if the QWSL miniband width is less than the optical phonon energy. This electron-optical phonon scattering mechanism becomes more important at higher temperatures.

Electric field domain formation is intrinsically related to the existence of the Wannier-Stark ladders and the so-induced negative differential conductance, and is believed to be even more detrimental in destroying BO. To overcome these technical obstacles, we propose to generate BO in QDSL. A QD is a semiconductor analog of an "atom." Inside a QD, the motion of an electron is confined and its energy is quantized into a series of eigenstates. This complete quantization is very similar to an electron in an atom.

When many QDs form a periodic crystal lattice, or QDSL, the coupling between dots leads to the formation of coherent bonding-antibonding states, and thus the so-called Bloch minibands. When biased with an external electric field, a miniband transforms into even-spaced Wannier-Stark ladders and BO is generated.

In contrast to QWSL, in QDSL, by varying the strength of electric field and its orientation with respect to the crystal axes, it is possible to suppress completely the electron-optical phonon scattering. As a result, the BO damping time, even at room temperature, may exceed the BO period by several hundred times. Furthermore, in QDSL, varying the coupling strength between two quantum dots allows us to finely tune the junction resistance. A finite junction resistance can be used as the shunt resistance, and may help to prevent formation of electric field domains.

FY 2006 Accomplishments

We observed the expected negative differential conductance (NDC) in the nonlinear I-V measurements. In two samples of different electronic potential modulations (3 percent vs 15 percent), the I-V trace is ohmic at small biases and shows an NDC behavior at higher biases. The observation of NDC is encouraging since it may represent the first step toward the establishment of electron self-oscillations in our QDSL. More importantly, the I-V curve displays different characteristics in two samples. This clearly shows that the I-V characteristics, and probably the 2D electron dynamics, can be manipulated in a controllable way.

In order to understand the physical origin of NDC in QDSL, theoretical simulations have been carried out using a one-dimensional modulation model. Except for this, other parameters are chosen according to a real sample. Assuming Bragg scatterings for the 2D electrons at the boundary of the Brillouin zone and a single life time for all the electrons, the theoretical I-V curve shows good agreement when compared to the experimental results.

We obtained more evidence of BO in magneto-transport experiments. Under high source and drain biases (Vdc), we observed a resonance-like resistance spike. The value of the magnetic (B) field where the spike occurs varies as Vdc is changed. In fact, when plotting Vdc vs B, a relationship of Vdc ~ 1/B is observed. At the present time, we believe that this 1/B dependence is due to the so-called edge-magnetoplasmon resonance (EMP). EMP resonance has been observed before in QD array samples but under external high-frequency radiation. In comparing with previous experiments, we conclude that in our QDSL experiment, the required high-frequency radiation is probably due to the electron self-radiation, i.e., BO.

We fabricated samples with different pitches and depths using the state-of-the-art interferometric lithography technique developed at the Center for High Technology Materials at the University of New Mexico. For future optical measurements, we also fabricated samples with semitransparent metal grids and with no metal grids.

We studied the quantum physics under extreme conditions in QDSL. At very low temperatures and high magnetic fields, we observed fully developed quantum Hall states at the Landau level fillings nu = 1, 2….. What's interesting is that in QDSL, the strength of the nu = 1 state is significantly reduced. This observation is a total surprise and vastly different from a bulk and unpatterned sample where the nu = 1 quantum Hall state is very strong. We believe this weakening of the nu = 1 state is due to a possible spin texturing state at the edge of the quantum Hall ferromagnets, which may have important implications in future quantum computation.

Our findings are important for an ultimate direct demonstration of BO in QDSL and extremely well suited to Sandia's unique expertise in RF electronics and quantum nanoelectronics. Scheduled milestones are complete, and remaining milestones are on track.

Significance

BO was originally proposed for electrons in a 3D periodic solid in 1928. Since then the topic has attracted a lot of experimental and theoretical investigations. The main motivation behind the research of BO is its huge potential in device applications; the BO frequency can fall into the terahertz region when the magnitude of electric field and the period of potential are carefully chosen. Compared to the existing terahertz emitters and detectors, for example, the quantum cascade lasers, the biggest advantage using BO is frequency tenability.

The Bloch frequency is determined by two parameters: the lattice constant of periodic structures and the external electric field, i.e., f = edE/h. It is clearly seen that in a device with a lattice constant of 350 nm, the Bloch frequency can be easily tuned from ~ 100 GHz, in the region of millimeter wave with an electric field of 1 kV/m, to ~ 1 THz, and in the terahertz regime with an electric field of 10 kV/m. Unlike other terahertz sources where the frequency is generally fixed for a single device, this wide tenability is the biggest advantage in using BO as a terahertz emitter and detector.

The proposed device structure, the 2D QDSL, is totally new and previously untested. As pioneers in this field we, for the first time, demonstrated in QDSL the negative differential conductance and edge magnetoplasmon resonance-like behavior under large electric fields. These two results, we believe, represent the first convincing evidence of electron self-oscillation in a QDSL structure. Once confirmed in the planned spectral measurements, we expect that these results could lead to ultimate realization of frequency-tunable, solid-state terahertz emitters and detectors.

This project is synergistic with several other ongoing activities at Sandia, including work in high-mobility 2D electron layer growth, quantum electron transport, and coupled electronic and photonic nanostructures. This project will draw on Sandia's expertise in quantum transport and high-frequency measurements, molecular beam epitaxy growth, and large-area nanometer-scale patterning, developed through those other, more fundamental projects.

In addition, results of this project will impact several ongoing applied projects that would benefit from demonstrated BO devices. These include projects on terahertz quantum cascade lasers, as well as on novel terahertz detectors using electron plasmon resonances, electron percolation in disordered potentials, and quantized conductance steps in ballistic one-dimensional wires as detection mechanisms.

BO devices with enhanced performance and/or multifunctionality could be integrated with other terahertz devices to produce terahertz subsystems, such as transceivers, with new characteristics. This project would thus complement other work and further cement Sandia's reputation as the originator of novel terahertz technologies that cannot be obtained elsewhere. We believe that this work will continue to buttress Sandia's position as a leader in this technology area.

Other Communications

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W. Pan, R.G. Dunn, J.L. Reno, J.A. Simmons, D. Li, S.R.J. Brueck, and S.K. Lyo, "Anomalous Electronic Transport Features in a Lateral Quantum Dot Array Sample," presented at the American Physical Society March Meeting, Baltimore, MD, March 2006.

W. Pan, R.G. Dunn, J.L. Reno, J.A. Simmons, D. Li, S.R.J. Brueck, S.K. Lyo, "Evidence of Bloch Oscillation in Lateral Quantum Dot Superlattice," presented at the NSF National Nanotechnology Infrastructure Network Annual Review Meeting, Austin, TX, February 2006.

Inverted Monolithic Interconnected Module (MIM) Thermophotovoltaics (TPV) for Remote Power Generation

93512

E. J. Heller, T. S. Luk, J. E. Strauch, J. D. Blaich, J. G. Cederberg, S. R. Lee, B. L. Silva, G. Girard

Project Purpose

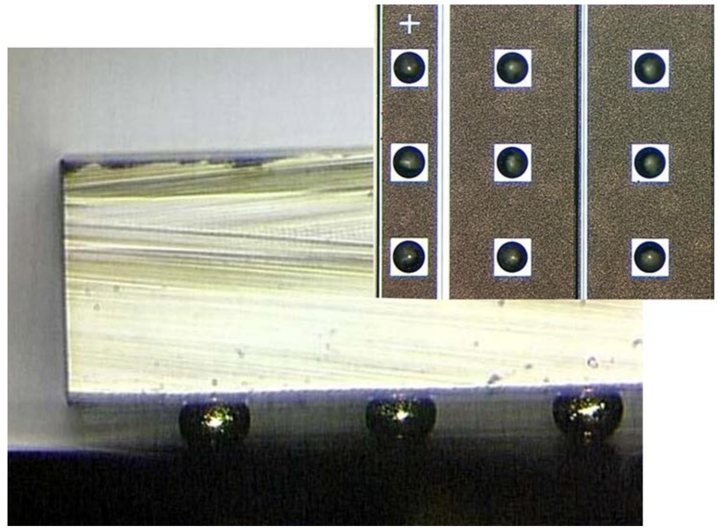
This purpose of this project is to increase thermophotovoltaic (TPV) monolithic interconnected module (MIM) efficiency (20 percent is the state of-the art) to enhance its potential for use in small, portable power supplies. The standard MIM design has several advantages over single junction devices:

- It decouples electrical and optical design.
- It reduces dark current resulting in higher output voltage.
- It allows for direct drive of logic circuits at 3-5 volts by building voltage on chip through series connections.

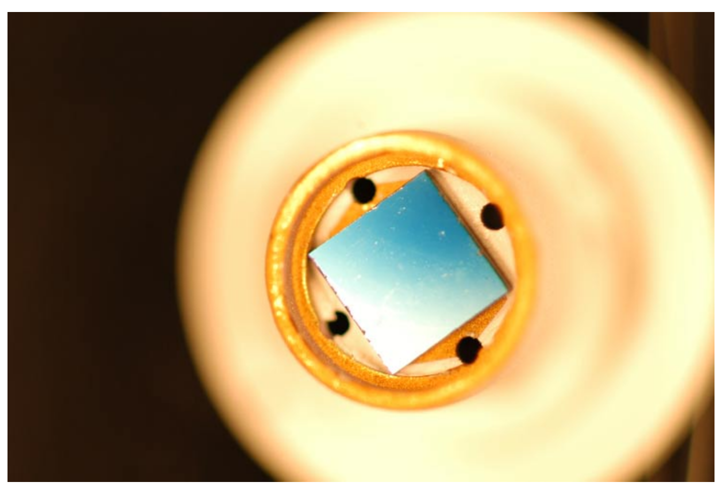
Further advantages can be obtained through a novel, inverted MIM design combined with Sandia's three-dimensional (3D) tungsten photonic crystal (PC) technology. The inverted design allows illumination through the infrared transparent InP substrate, eliminating shadowing due to topography or grid fingers. This embodiment also will allow deposition of an integrated spectral control (optical) filter directly on the planar back surface.

The spectral control filter reflects light that is too low in energy to produce electron-hole pairs (i.e., nonconvertible light) and thus simply heats the cell, degrading its performance. The integrated filter reduces the volume required when compared to the normal glued-on filter approach, an important aspect in power microsystems.

The TPV cell uses mismatched epitaxial growth of InGaAs on an InPAs buffer on a semi-insulating InP substrate. The inverted MIM design not only decouples the electrical and optical characteristics of the cell, but also reduces the thermal impedance, and incorporates a diffuse back surface reflector trapping the convertible light within the cell. If trapping the convertible light is successful, the base thickness could be reduced, creating an inherently more radiation-hard cell. This is helpful when using a radioactive heat source.



Side view of a completed flip chip inverted thermophotovoltaic monolithic integrated module device with under-bump metallurgy. Inset shows a solder bump array before flip chip mounting. The solder bumps in the upper right inset are 150 microns in diameter. The wafer thickness in the main picture is 610 microns.



A flip chip inverted thermophotovoltaic monlithic integrated modeule mounted on a boron-nitride coated aluminum header for electrical testing. Note the lack of structure and grid line shadowing on the illuminated surface.

Further spectral control is achieved using 3D tungsten PC emitters developed at Sandia, which tune the spectra incident on the cell. Modeling indicates that the development of the novel inverted MIM design combined with Sandia's 3D PC tungsten technology could lead to 28 percent conversion efficiencies.

The objectives for the first year of the project were focused on developing the MIM design, growth, processing, and characterization capabilities. Integration of the PC emitter with the MIM and testing in a simulated thermal environment is planned for the second year. The third year objective is to incorporate an optimized MIM/PC into a complete TPV power generation system. This unique design, which relies on the epitaxial growth capabilities of Sandia combined with our pioneering PC technology, will enable a remote power source which exceeds any currently available system.

FY 2006 Accomplishments

We completed all first-year milestones.

We designed a strain-relaxed InPAs buffer for a lattice mismatched 2.1 micron InGaAs device on a semi-insulating InP substrate, and grew it using metal organic chemical vapor deposition epitaxial growth. We then performed x-ray diffraction reciprocal space mapping using a position-sensitive detector to evaluate the strain and composition of InPAs and InGaAs layers. Orthogonal arrays of misfit dislocations aggregated and formed a "cross-hatch" pattern with a characteristic roughness.

We achieved reproducible doping of InPAs and InGaAs with Si and Te (n-type), or Zn (p-type) to levels in excess of $1x\ 10^{19}$ per cubic centimeter, a level allowing low-resistance contacts and tunnel junctions for inverting the polarity of the device. Te-doping of InPAs reduced the surface roughness associated with the InPAs buffer cross-hatch pattern, suggesting it enhances surface mobility during epilayer growth.

InGaAs wafers (both from Emcore and in-house) were fabricated into both front and back-side illuminated (inverted or flip chip) TPV cells and MIMs. We developed and demonstrated an under-bump metallurgy (UBM) layer process to allow flip-chip bonding of the backside illuminated version.

We measured maximum output power at 1.0 W/cm^2 and 4.2 A/cm^2 short circuit current density (J_{sc}) at 300 K. The 6-junction device has good open circuit voltage (V_{oc}) of 2.36 V, or 394 mV per junction, and a fill factor (FF) of 68.9 percent. The FF, V_{oc}, and J_{sc} for this device are quite respectable for early results on in-house-grown material. Reductions in the defect density from the Sandia growth process should enable us to match the performance of the Emcore production wafers.

We initiated integrated testing in vacuum of 3D tungsten photonic crystal (PC) emitters and TPV cells. The components used for these tests permit emitter temperatures of 1400 K while maintaining 300-400 K TPV cell temperature. The photonic crystal, developed at Sandia, is a spectral emitter designed to selectively emit light of 1-2 microns wavelength, which is optimum for the TPV device with a band-edge of 0.6 eV or 2 microns. In support of these integrated tests, we developed packaging methods to mount TPV cells to both aluminum and aluminum nitride substrates. Integrated testing has demonstrated 1400 K photonic crystal temperature while maintaining less than 350 K at the TPV cell.

Significance

This thermophotovoltaic approach adds another tool for converting heat into useful electrical power, joining more conventional approaches based on thermoelectric (TE) materials. Large-scale TE converters can approach 3 percent to 5 percent efficiency in converting thermal energy into electrical power. However, in miniaturized systems the efficiency falls to less than 1 percent. Furthermore, additional circuitry is typically required to boost the voltage of a TE converter up to a useful level, lowering the system efficiency even further.

The thermophotovoltaic approach promises much higher efficiency (current diodes are about 20 percent efficient), and makes it easier to build voltage on chip without needing additional power- management circuitry. These unique benefits of TPV technology make it an attractive energy-harvesting method. Furthermore, the TPV approach is readily scalable from milliwatts to hundreds of watts.

Thermophotovoltaic technology has been pursued by a relatively small community, primarily for space applications. While showing promise, it still has not been able to displace more traditional approaches, so development has been slow. Sandia has strong interest in the technology for autonomous sensored microsystems, a niche not of interest to other developers. This may indeed become the first area where TPV gains wider acceptance.
This project positions Sandia quite strongly within the TPV community, having all aspects of a credible effort from crystal growth through design, fabrication, and testing. This position will enable us to guide and speed up the development of the technology for our missions.

Other Communications

J.G. Cederberg and S.R. Lee, "*Optimization of Strain-Relaxed InPAs Buffers for InGaAs Thermophotovoltaic Devices*," to be published in a Sandia SAND Report.

A Discovery Platform for Nanowire Electronics and Photonics

93513

A. A. Talin, L. L. Hunter, E. Marquis, G. T. Wang, J. R. Creighton, J. W. Hsu, J. G. Fleming

Project Purpose

The purpose of this project is to create a platform for integration and characterization of semiconductor nanowires. One-dimensional (1D) nanostructures such as carbon nanotubes and inorganic nanowires present distinct and desirable properties stemming from their anisotropic dimensions and carrier quantum confinement. Potential applications include:

- ultraviolet (UV)/visible/infrared (IR) nanolasers and waveguides for nanophotonics and biochemical microanalysis
- room temperature ballistic conductors for high-frequency/high-power circuits
- sensors for biochemical agents
- single electron transistors for ultralow power circuits.

Many research groups have demonstrated a large variety of nanowires using either liquid solution synthesis or gas phase deposition on solid substrates using the vapor-liquid-solid reaction. Despite these advancements in the basic synthesis and growth, straightforward electrical and optical characterization of these quasi 1D nanostructures and their integration with conventional Si circuitry for device applications have remained challenging.

This project addresses these challenges in three steps: 1) development of phase-I platform for postgrowth assembly and optical/electrical characterization of nanowires; 2) using a top-down technique (nanoimprint lithography) to create dense, uniform, large-area arrays of semicon-ductor nanowires; and 3) providing a phase-II platform for large-scale vertical integration of nanowires.

FY 2006 Accomplishments

Phase I Platform

We developed a platform for individually contacting a large number of semiconductor nanowires grown by either vapor-liquid-solid (VLS) or solution synthesis routes. It consists of arrays of individually addressable interdigitated electrodes with micron gaps patterned over nanowires that are randomly dispersed on Si/SiO_2 substrates. For characterization, we interfaced a focused He-Cd laser with a probe station. Using this platform we are able to electrically/optically characterize tens to hundreds of nanowire devices on a given wafer and correlate results with growth conditions. We demonstrated that increasing GaN nanowire growth temperature from 800 ºC to 900 ºC increases conductivity by 10^5 and significantly improves photoluminescence.

Phase II Platform

We fabricated the first substrate for nanowire vertical integration using nanoimprint lithography (NIL). Using Au catalyst, this platform was successfully used for ordered growth of Ge nanowires. Using NIL and silicon-on-insulator (SOI) substrates, we fabricated and characterized a large, uniform array of Si nanowires using a strictly top-down technique. The electrical transport measured in these lithographically defined nanowires is useful for comparison to chemically synthesized nanowires, which may contain impurities.

Significance

The rapid increase in performance of microelectronics, as often summarized by Moore's law, is arguably responsible for the information revolution that has so far characterized the end of the 20th and beginning of the 21st century. To continue along this trend, however, the IC (integrated circuit) industry faces two daunting challenges:

- the enormous rise in manufacturing costs associated with fabrication of transistors with sub-100 nm critical dimensions, and
- the fundamental design limits to the traditional planar Si metal-oxide-semiconductor field-effect transistor (MOSFET), which lead to unacceptable gate and source-drain leakage currents, heat dissipation problems, and ultimate failure of the transistor as a switching device.

To address these issues, new and innovative solutions are needed that depart from the traditional top-down fabrication techniques that have thus far dominated IC fabrication and that are now running into what many in the industry call the 'red-brick wall.' In addition, new semiconductor device physics that incorporates first principles treatment of carrier transport, contact formation, and electron-phonon interactions has to be developed to better represent and understand nanoscale device behavior. The work and results of this project will impact the above challenges by providing a route to introduce novel growth techniques and materials that will enable future circuits to be faster, denser, and less power consuming.

These advances will help our nation keep its technological edge in the future. The relevance to Sandia's mission is two fold: first, there is always a need for higher performance and lower power consuming circuitry; second, in-house expertise is essential for understanding how new electronic components will behave in the various applications and environments encountered relevant to Sandia.

Refereed Communications

A.A. Talin, L.L. Hunter, F. Leonard, and B. Rokad, "Large Area, Dense Silicon Nanowire Array Chemical Sensors," to be published in *Applied Physics Letters*.

F. Leonard and A.A. Talin, "Size-Dependent Effects on Electrical Contacts to Nanotubes and Nanowires," *Physical Review Letters*, vol. 97, pp. 026804-1 - 026804-4, July 2006.

Other Communications

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G.T. Wang, J.R. Creighton, A. Talin, E. Lai, I. Arslan, D. Werder, and P. Provencio, "MOCVD Synthesis and Characterization of III-Nitride Nanowire and Heterostructure Nanowire Arrays," presented at the Materials Research Society, San Francisco, CA, April 2006.

E. Lai, A.A. Talin, G. Wang, R. Creighton, R. Anderson, and L.L. Hunter, "Effect of Growth Temperature on the Electrical and Optical Properties of GaN Nanowires," presented at the Materials Research Society, San Francisco, CA, April 2006.

Miniature Flow Cytometer for Medical Diagnostics and Pathogen Detection

93515

I. Brener, S. K. Ravula, D. W. Branch, J. B. Wright, W. C. Sweatt, C. D. James

Project Purpose

A conventional flow cytometer uses hydrodynamic focusing to generate a narrowly focused (~ 10 μm) diameter sample stream moving with m/s linear velocities that is interrogated by a tightly focused laser beam (10-100 μm in diameter). Light scatter, along with spectral bands of fluorescence, is collected from micron-sized particles (cells or microspheres) using very sensitive detectors (photomultipliers or avalanche photodiodes). With careful design, flow cytometers can detect as little as a few hundred fluorophores at conventional analysis rates and single fluorophores with reduced analysis rates.

The high linear velocity and small interrogation volume result in transit times of a few microseconds, which allows multiparameter observation of population distributions and provides continuous kinetic resolution at analysis rates of > 10,000 per second. To achieve this high analysis rate, conventional flow cytometers use high-speed data acquisition components (running at frequencies up to 20 MHz) to record the very short pulses derived from the fluorescence and scatter signals.

Several attempts have been made to create portable flow cytometers. However, these instruments are still far too power hungry (> 24 W) and large (the smallest weighs ~ 30 pounds without battery or computer). Moreover, the limited performance of some of these instruments in terms of excitation wavelength, detection wavelengths, and sensitivity make them ill-suited for analysis of soluble microsphere arrays, which requires at least three fluorescence channels. Finally, all current flow cytometers require large volumes (liters/day) of sheath fluid, which significantly limits the portability of such instruments.

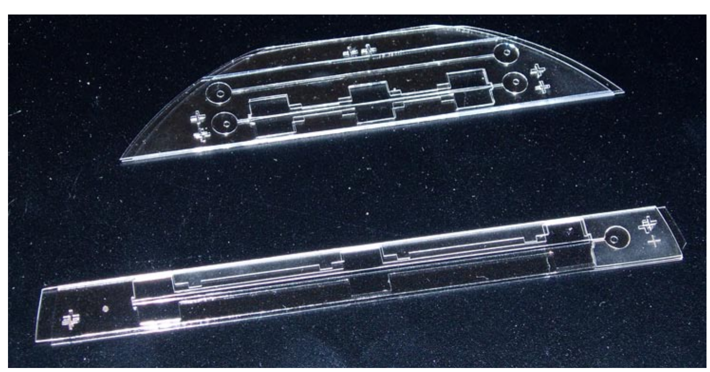
The goal of this project is to develop the technology necessary to miniaturize a sheathless flow cytometer, based on acoustic focusing in microchannels instead of the more conventional hydrodynamic focusing used in cytometry flow cells. Our efforts at Sandia concentrate on a microfluidic version of this device, i.e., acoustic transducers integrated with microchannels and a micro-optical train.

FY 2006 Accomplishments

- Fabricated thin film piezoelectric transducers on Si for acoustic focusing in microfabricated microchannels, with high d33, and high yield of large (1 mm x 30 mm) devices.
- Performed routine measurements of piezoelectric properties of the thin lead zirconate titanate (PZT) transducers and preliminary measurements of acoustic levitation using these films.
- Performed preliminary acoustic focusing in thin-walled capillaries
- Fabricated Foturan microchannels for side PZT transducers using direct photopatterning and wafer bonding. The PZT transducers need to be inserted sideways to the flow direction to permit top/bottom optical access.

Significance

We developed the capability to fabricate microfluidic channels with embedded piezoelectric transducers. These devices will be a key component of this and other platforms for chemical and biological sensing, both for civilian and military use. These devices can also be used for other applications that require microparticle manipulation (beads, cells, and so on), and can be leveraged in other sensor devices.



Picture of Foturan channels with slots for PZT transducers.

Just in Time Jamming of Enemy Detonation Signals

93516
L. D. Bacon, W. S. Pickens, G. A. Wouters, R. W. Brocato, D. Greenway Jr.

Project Purpose

Our primary goal is to develop a prototype, man-portable, reactive radio frequency (RF) countermeasure for radio-controlled improvised explosive devices (RCIEDs). Our focus this year has been on the critical technology, which is the real-time spectrum analyzer (RTSA) aspect of the receiver. We completed the system and key component design, fabricated and tested the key microcomponents, and performed initial system integration experiments.

FY 2006 Accomplishments

System Topology
We identified a system topology that we believe will enable our goals. This topology is a hardware real-time spectrum analyzer (RTSA). Our testing of target communications devices of interest indicates that a hardware approach like the one we are taking is necessary to achieve the response time/power consumption requirements for a man-portable JITJ (just in time jamming) system. We selected a 16-channel RTSA that would defeat a difficult target of interest as our proof-of-concept prototype.

Key System Components
Based on this topology, we identified the key system components: high-Q surface acoustic wave (SAW) filters and multimode or active power dividers. Both of these critical components use unique Sandia skills and processes in their design.

System Integration
We selected the commercial-off-the-shelf low-noise amplifiers (LNAs) for the prototype system and have begun testing the layout of the critical sections of the receiver board.

Benchtop Trade-Off Testing
We were able to leverage information from a complementary project that takes a software approach to signal detection – as opposed to the microelectronics hardware approach taken here – to accomplish the system trade-off experiments. This allowed us to focus on the critical components of the system. We are well ahead of schedule on this most-important aspect of this project.

Significance

We developed a unique capability to produce very high narrow-band SAW filters that can be placed precisely on frequency. In addition, we designed and produced low-loss broadband splitters. Thus, we designed, built, and tested the major critical components that will enable us to produce a very fast-response prototype solution to counter the use of RCIEDs.

Ultrasensitive Directional Microphone Arrays for Military Operations in Urban Terrain and Future Combat Systems

93518

M. Okandan, E. P. Parker

Project Purpose

Through this project we aim to develop extremely high-fidelity directional microphones that will have applications in acoustic source localization and identification. We demonstrated interferometric sensitivity (usually obtained by benchtop systems, on the order of 10^{-15} meters) in a micromachined device assembly (~ 1 cm^3). This level of sensitivity is critical for enabling the overall system goals for directionality and resolution. With ultimate performance and final system cost metrics in mind, we are investigating different assembly techniques for further integration of the system components.

The main advantage of the system under investigation is the optical sensing mechanism that is used instead of capacitive sensing, which is more common in microscale sensors. The size and sensitivity metrics scale in opposite directions for capacitive sensing (smaller electrode area means less signal), but for optical sensing, sensitivity and size are decoupled. This also opens up the simultaneous electrostatic actuation capability for the optical sensing setup without the inherent complications encountered in capacitive sensing approaches.

FY 2006 Accomplishments

- Developed the most advanced microphone damping model demonstrated to date, which employs a finite element squeeze film damping analysis of the microphone backplate. The model has been used to explore the new design-space offered by the optical detection structure so that extremely low thermal-mechanical noise levels can be realized with fabricated designs.
- Designed and microfabricated optical microphone array structures with individual array elements with 20 kHz bandwidth for extreme sensitivity applications and greater than 50 kHz bandwidth for broadband fingerprinting applications.
- Rigorous frequency response characterization of fabricated microphone structures demonstrating bandwidth, damping, sensitivity, and thermal-mechanical noise levels within five percent of design targeted values.
- Developed Fourier transform-based near-field diffractive optics model for optoelectronic integration design of a sub 1 mm3 fully integrated microphone employing vertical cavity surface emitting laser (VCSEL) technology.
- Fabricated and tested custom VCSEL + photo-detection structures on GaAs (fabricated in Sandia's Compound Semiconductor Research Laboratory (CSRL)).
- Integrated prototype of custom GaAs with advanced microphone structures and demonstrated functioning optical microphone elements in a volume of less than 1 mm3.

Significance

Devices being developed in this project are opening up new application areas for acoustic sensing with their unprecedented performance, size, and power consumption metrics. This project is in line with Sandia's aim to pursue projects and technologies that Sandia can uniquely address with its diverse expertise and infrastructure. We engaged extensive expertise and resources from around Sandia to enable rapid progress on this project.

Refereed Communications

N.A. Hall, M. Okandan, and F.L. Degertekin, "Surface and Bulk-Silicon-Micromachined Optical Displacement Sensor Fabricated with the SwIFT-Lite™ Process," *Journal of Microelectromechanical Systems*, vol. 15, pp. 770-776, August 2006.

Other Communications

N.A. Hall, M. Okandan, and F.L.Degertekin, "Finite Element Modeling of Thin-Film Damping in Micromachined Microphones Employing Diffraction-Based Optical Readout," to be published in *Acoustical Society of America*.

High-Power Broadly Tunable Mid-IR Quantum Cascade Lasers for Improved Chemical Species Detection

93519

E. W. Young, J. F. Klem, C. T. Fuller, M. A. Mangan, M. C. Wanke

Project Purpose

The purpose of this project is to examine a novel miniband laser design that should inherently increase output power while providing a broad tuning range. These novel high-power quantum cascade lasers should be able to tune greater than 10 percent of their center mid-infrared frequency. This technology will enable multiple chemical species identification with a single laser and/or very broad frequency coverage with a small number of different lasers, thus reducing the size and cost of chemical detection systems.

FY 2006 Accomplishments

We created new code that automates calculation of miniband quantum cascade structures based on predetermined design rules and desired parameters. This code was successfully integrated into our existing modeling software, greatly enhancing our future design capabilities. The improved software will reduce the turnaround time and increase consistency between designs for miniband structures, as well as provide enhanced, quick, and economical designs of a variety of other quantum cascade lasers.

We designed, fabricated, and tested our novel mini-band quantum cascade laser structure. However, due to issues of poor growth quality, the results thus far have been inconclusive. We performed a second round of growth of the original structure, as well as several variations of the original structure. Processing of the new material is ongoing at the time of this report.

Significance

The improvements made to our modeling software allow us to provide enhanced, quick, and economical designs of a variety of quantum cascade lasers. This means that temporal and monetary costs will be reduced for any future projects requiring the design of quantum cascade laser structures. This is especially critical when iterating a design concept, as the software improvements increase consistency and turnaround time.

Though the feasibility of our novel laser design concept has yet to be proven due to lack of good quality material, if successful this technology will enable multiple chemical species identification with a single laser and/or very broad frequency coverage with a small number of different lasers, thus providing Sandia laser sources capable of reducing the size and cost of chemical detection systems for national security and industrial applications.

Si-Rich Silicon Nitride Films for Reliable Low-Write Voltage Antifuses

93520

D. J. Stein, R. D. Nasby, A. H. Hsia, S. D. Habermehl, R. T. Apodaca, E. Roherty-Osmun

Project Purpose

We propose to develop special films that enable integration of antifuses (AF) with complementary metal oxide semiconductors (CMOS). User-selectable write voltages of 5 volts or less will ensure congruence with the CMOS technologies in Sandia's Microelectronics Development Laboratory (MDL). Antifuses are write-once-read-many memory devices that allow post-fabrication programming of read-only memory.

Currently, antifuses are built with complex film stacks of thin amorphous-Si, Si_3N_4, and SiO_2. Besides the inherent difficulty in controllably depositing the extremely thin films of these materials (thin is necessary for a low-voltage write) and the associated manufacturing complexity, these films exhibit a broad distribution of write voltages centered around 8-9 volts, which is outside the normal operating voltage of MDL's CMOS.

Our specialized films can be tailored to break down in lower electric fields and have more consistent breakdown characteristics than SiO_2, thus they make good candidates for an AF dielectric. Existing data shows that one of our specialized films breaks down at 4 volts (2 MV/cm). We developed film deposition techniques and investigated various film compositions, and started integration development. In the second year, we will optimize the integration scheme and perform reliability assessments.

FY 2006 Accomplishments

- We developed deposition techniques for high-temperature CVD-deposited (chemical vapor deposition) and low-temperature plasma-deposited AF films. The films demonstrate write voltages between 1 and 10 volts depending on composition. The plasma process required extensive process development. We tried two approaches and, after significant development work, settled on a favored one. In addition, the process to deposit the 100-200 Å thick dielectric is performed in a tool designed to deposit at 700 nm per minute. Control of thickness and uniformity across the wafer took several tool modifications, including changing mass flow controllers, wafer heat-up time, and radio frequency settings.
- We tested and verified no degradation of existing CMOS materials under the deposition conditions of the high-temperature CVD-deposited AF film (850 ºC).
- We designed potential integration schemes for these films and designed and placed into a reticle set a series of technology characterization vehicles to build testable structures.
- We tested short-loop integrated AF structures. For the low-temperature plasma-deposited AF dielectric, we successfully demonstrated working antifuses in a short-loop lot. This lot had seen silicidation, pre-metal dielectric deposition, contact formation, metallization, and passivation.
- We began fabricating short-loop lots to investigate the high-temperature CVD-deposited AF dielectric.

Significance

Potential applications of this AF technology include trimming, unique addressability, secure applications, serialization, and manufacturing simplification. Existing projects that would benefit from AF include ViArray and UXI/Shaman. ViArray is a generic design that contains all of the aspects of ASICs (application specific integrated circuit). ViArray is designed to support a variety of customer applications by specially tailoring only the layout of via2 for a standard design rather than designing an ASIC for each application from scratch. Many customers desire long-term, nonvolatile memory component in their parts. Our AF technology should satisfy this requirement without extensive changes in our current CMOS process.

Rapid Spectroscopy for Gas Cloud Analysis

93521
M. C. Wanke, E. A. Shaner, J. L. Reno, M. Lee, W. J. Zubrzycki, A. D. Grine, J. D. Williams, C. T. Fuller

Project Purpose

Spectroscopic signature measurements show that terahertz frequencies offer unprecedented capabilities that conventional microwave and infrared wavelengths don't have. We are exploiting these new findings by using Sandia's existing strengths in terahertz microelectronics and microsensor packaging to prototype a sensor platform capable of detecting terahertz spectra for various potential applications. This sensor is based on an innovative new microelectronic terahertz detector developed at Sandia. The sensor, which can easily be multispectral, will operate at known spectroscopically relevant terahertz wavelengths and have sufficient speed/bandwidth to follow submicrosecond events.

Previously Sandia has focused on the device physics and fabrication of terahertz detectors. In this project, we are focusing on integrating Sandia-developed terahertz detectors into a form that can be used in a compact terahertz sensor system. The initial devices are being used to expand the diagnostics for dynamic spectroscopy at our gas-gun facility from the visible and infrared into the terahertz region. The combined capabilities would offer a much broader range of material identification possibilities than achievable with the atomic measurements provided by visible and infrared spectroscopy alone.

Our gas guns constitute a unique test bed that provides well-controlled experimental environments that match operational conditions and therefore offers a realistic test of the detector capabilities.

FY 2006 Accomplishments

- Modeled the emission properties of a test scenario on the gas guns to determine the operational parameters needed (frequency selection and noise equivalent power (NEP)) for the detector array to reliably identify gas molecules in an expanding vapor cloud.
- Designed and started assembly of initial terahertz diagnostic to validate model results and determine whether terahertz can be used in specific operational scenarios.
- Fabricated a multipixel array with each pixel tuned to a different frequency when they are all biased with the same gate voltage.
- Developed test capability to measure the detector NEP to be able to model system performance. Determined that the NEP of the detectors is not quite good enough yet, requiring effort next year to improve the NEP first before trying to build a packaged system.

Significance

The major improvement of detector NEP and the development of multispectral pixels offer hope of a multispectral detector element for instantaneous spectroscopy over wide bandwidths. Unfortunately, the biasing requirements used to increase the NEP need to be simplified to take advantage of the promise in a meaningful way. If we manage to continue improvements in both the NEP and bias condition simplification, this device can become competitive with existing detectors for responsivity, offering high speed and narrow bandwidths.

The experimental measurements on the gas gun enable the first measurements on what the signatures of an event will really look like and that we can use to verify the various assumptions required for a theoretical analysis of the problem. Realization of this prototype system may spur investments from internal and external customers interested in exploiting the terahertz opportunity for numerous other applications.

Refereed Communications

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, "Tunable THz Detector Based on a Grating Gated Field-Effect Transistor," in the *Proceedings of the SPIE - Photonics West*, p. 612006-1, January 2006

Developing Key Capabilities for Quantum Computing

93522
L. G. Pierson, M. G. Blain, M. P. Lilly, O. B. Spahn, M. H. Crawford, M. A. Mangan

Project Purpose

The first nation to develop a quantum computer (QC) capable of breaking other nation's codes will have a great advantage over its adversaries. The physics of QC is sound, yet the engineering of QC for useful applications has yet to be done. Whether (and when) a QC can be designed, engineered, and scaled to the point of successful cryptanalysis of conventional codes is uncertain. With this project we aim to reduce that uncertainty by developing certain key hardware capabilities necessary for implementation and processing of quantum bits (qubits) and to make significant strides toward the realization of quantum simulation and other QC applications.

This effort is focused on demonstrating viability of methods of ion control, optical beam control, and compact laser beam generation necessary for implementation of qubits in trapped ion technology and viability of implementing qubits using vertically coupled quantum dots. Part of this work is to validate methods of electromagnetically transporting ions on microelectromechanical systems (MEMS) ion trap chips (ITCs). This transport is necessary for realization of a quantum gate.

In order to use these devices to perform real quantum computing, we are developing the ability to focus laser beams on the ions to perform ion cooling and to manipulate and read out quantum states by simultaneously controlling multiple optical beams. Currently, lasers of the correct wavelength to perform ion cooling, optical pumping, and manipulation of quantum states consume a large portion of an optical bench, and we are working to develop semiconductor lasers that operate in the needed ultraviolet (UV) wavelengths that are necessary to make operation of large ion arrays practical.

Finally, solid-state qubits are expected to evolve to the point of satisfying quantum operations even more efficiently than ions, and we intend to investigate a promising solid-state technology that may guide "next generation" quantum computing.

FY 2006 Accomplishments

Ion Trap Validation

We designed a first-generation ion trap validation system to test the operational characteristic of the trap assemblies. We integrated the first few components with a custom ion trap chip package, and the validation system is on track to examine the results of ion trap array fabrication and packaging efforts. Radio frequency and direct current electrical feedthroughs and connections in the vacuum chamber are near completion.

Micro-Optics

We derived a general set of requirements for the MEMS mirrors based on discussions with trapped ion research groups at the National Institute of Standards and Technology and at the University of Oxford. We designed and fabricated initial devices, and ordered and received most of the high-speed control electronics necessary for temporal evaluation of these devices. We completed parts fabrication and began to evaluate performance.

UV Sources

We performed bandstructure and many-body gain calculations for AlGaN quantum well structures. Results included the prediction of the wavelength dependence of peak spontaneous emission and peak gain as a function of carrier density, which is critical to optimizing active region designs for our 313 nm target lasing wavelength. Advances in our metal-organic vapor-phase epitaxy growth process for AlGaN device structures enabled threading dislocation densities of $9.5x10^8/cm^2$, meeting our milestone of $< 1x10^9/cm^2$ defect densities.

To achieve even further defect reduction, we investigated the growth of $Al_xGa_{1-x}N$ (x = 0.3 - 0.6, 1) epilayers using cantilever epitaxy (CE) on patterned

SiC substrates as well as growth on patterned structures in AlN or GaN templates. We achieved lateral growth and coalescence of AlN epilayers on patterned SiC substrates, and transmission electron microscopy analysis confirmed reduced dislocation densities in the regions of lateral growth.

Vertically Coupled Quantum Dots
We fabricated and measured two generations of single quantum dots in a single two-dimensional electron system. Measurement of the quantum state of electrons in quantum dots requires use of a sensitive electrometer in very close proximity to the quantum dot. We fabricated a multigate structure to place a quantum point contact very close to the opening of an electrostatically defined quantum dot. The quantum point contact will serve as a local electrometer where small changes in the conductance will occur as the charge state of the nearby quantum dot changes. With six independent gates controlling both the behavior of the dot and the quantum wire, control of the electrostatic potential becomes very complicated.

In the latter part of FY 2006 we focused on two additional objectives:

- building up an electronics system to fully characterize and control all of the gates and identify clean Coulomb oscillations as electrons hop on and off of the quantum dot
- minimizing the "telegraph noise" we detected in our coupled quantum dot/quantum point contact system.

We made progress in both areas and are moving on to measure single-charge events using both lateral and vertical geometries.

Significance

The Quantum Information Science and Technology Roadmap identifies technologies that hold promise for accomplishing the scalability, long decoherence times, initialization, manipulation, and measurement criteria needed to realize a quantum computer. Of these technologies, trapped ion approaches are closest to demonstrating the documented criteria in the near term, and eventually solid-state approaches (quantum dots, quantum wires, and so on) are expected to mature and interface more readily to classical electronic computer systems. This project is advancing both of these quantum computing device technologies.

Even though the processing of many thousands of qubits will eventually be required for cryptanalytic applications, the first few applications of quantum information processing will involve systems of dozens of qubits. The technologies being developed within this project are expected to enable a leap from the current state-of-the-art processing limited to 8 or 9 qubits in a "macro-ion-trap" to demonstrate processing with dozens of ions in a single "micro-ion-trap." Eventually, combination of multiple traps may enable far larger processing capability.

These goals require full characterization of micro-ion-trap assemblies, compact UV laser sources, micro-optical control of laser beams focused on the ions, and innovative control electronics to coordinate and integrate these components. The development of solid-state quantum processing technologies will augment this tool set and enable the efficient interface of specialized quantum processors to conventional electronics.

Our progress in achieving reduced defect density AlGaN epilayers may yield improved performance in a range of device structures, including solar-blind photodetectors, UV light-emitting diodes, and UV laser diodes. These devices can be applied to several applications of relevance to Sandia missions, including fluorescence-based biosensing and covert communications, as well as trapped ion quantum computing.

Development of integrated quantum point contacts for charge detection is critical for semiconductor implementations of quantum computing. Our recent efforts are pioneering this technology at Sandia.

Other Communications

M.H. Crawford, S.A. Allerman, A.J. Fischer, K.H.A. Bogart, S.R. Lee, W.W. Chow, M. Carroll, M. Banas, and J.J. Figiel, "Recent Advances in UV/Blue Laser Diodes and Detectors," presented (invited) at the DTO Workshop on Trapped Ion Computing, Boulder, CO, February 2006.

Controlled Synthesis of Nanocrystalline Catalysts from Solutions to Supports

104111
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Project Purpose

The purpose of this project is to develop a general method for nanocrystal synthesis to afford rational size (diameter) control and narrow size distributions for a wide range of materials. While methods exist for generating specific-sized nanocrystals, these are empirically-derived "trial and error" techniques that work for specific materials only. A general, rational synthetic strategy for controlling nanocrystal diameters and producing narrow diameter distributions has not yet emerged.

The synthetic method developed in this project is based on a new kinetic and mechanistic analysis of solution-based nanocrystal growth by aggregative processes for gold and bismuth. This method is of broad utility and applicable to a wide range of nanocrystal materials, including metals, metal alloys, ceramics, and semiconductors. No other available synthesis approaches the generality of this method. This technique will have a significant impact on nanoscience, removing limits to synthetic processes currently in use and allowing more rational syntheses of nanomaterials, thus removing manufacturing barriers to the controlled synthesis of nanocatalysts, semiconductors, metals, and the like.

The specific goals of this project are to:

- Survey and develop Pt-nanocrystal syntheses for systematic variation of nanocrystal size while maintaining narrow size distributions over a range of catalytically relevant diameters (ca. 2-10 nm)
- Identify preparative pathways and controlled passivation strategies to allow quantitative kinetic analysis of nanocrystal growth
- Look for and promote aggregative-growth mechanisms.

FY 2006 Accomplishments

We established two synthetic techniques to control platinum nanocrystal size: the phase-transfer synthesis and the polymer-stabilizing synthesis. These techniques involve the reduction of platinum in various complexes to metallic platinum. We were interested in routes to prepare Pt nanocrystals that did not also simultaneously produce significant amounts of bulk, reduced Pt metal.

We found the phase-transfer method to initially yield small, Pt nanocrystals in the 2 nm range, using tetraoctylammonium bromide (TOABr) and dodecylamine (DDA) as stabilizers. Growth of these nanocrystals could be induced thermally in appropriate solvents by adding various concentrations of additional TOABr.

We identified boundary TOABr concentrations that distinguish three different growth regimes in the phase-transfer process and clearly establish the concentration range necessary for the desired aggregative-growth mechanism. We found that:

- Low concentrations of TOABr lead to growth by Ostwald ripening, growing the 2 nm crystals to about 3.5 nm.
- Intermediate TOABr concentrations yield controlled aggregative growth with nanocrystal coalescence – a desirable growth mechanism – producing nanocrystals up to 6 nm in size.
- High TOABr concentrations lead to aggregation but not coalescence, producing aggregates of primary nanocrystals.

We found the polymer-stabilizing synthesis to proceed via a rapid generation of small primary Pt particles, which subsequently undergo aggregative growth, affording larger, narrowly dispersed Pt nanocrystals. This procedure uses a platinum(II) acetylacetonate complex, which is reduced by octadecanethiol (ODT) in the presence of a polymer stabilizer, polyvinylpyrrolidene-co-hexadecene (PVP-co-PHD).

- Time-dependent studies showed primary particles in the 1.5 nm range that undergo aggregative growth to 5-6 nm (20 minutes) to 8 nm (2 hours) to 9 nm (4 hours).

- We obtained kinetic profiles, revealing an initial induction period, followed by rapid aggregative growth, and then very slow Ostwald ripening.

Very slow Ostwald ripening is a synthetically useful outcome, because it indicates that the ultimate nanocrystal size will be determined by the aggregative-growth regime, which is amenable to chemical control.

We achieved our goal to control the particle size of Pt nanocrystals, and we used an aggregative-growth mechanism to explain the results, and we added nanocrystalline Pt to the list of metals that can grow in a controlled fashion by this mechanism.

Significance

We demonstrated that platinum nanocrystals can be grown in a controlled and predictable manner via aggregative growth. This result, along with previous results for bismuth and gold, indicates that controlled growth of nanocrystals in general is an achievable goal in the near future, and applicable to many systems.

Although not yet fully developed and optimized, the early indications for this growth method strongly suggest it will afford narrow size distributions and controlled Pt-nanocrystal diameters in the catalytically relevant size range of 2-10 nm. Additional research to attach these nanoparticles to catalytically important porous supports is needed to take maximum advantage of this work. Extension to other systems of interest, such as alloys, semiconductor materials, and other metals, should also be carried out.

Materials Sciences

Electrochemically Switchable Materials for (Bio)Microfluidics

67074
K. R. Zavadil, M. Farrow, B. C. Bunker, D. L. Pile

Project Purpose

Homeland security needs demand the development of a new generation of microfluidic devices that can concentrate, separate, and sense a wide variety of viral, bacterial, and pathenogenic agents. A key enabler of this technology is a class of functional materials capable of being programmed to perform operations on complex biological samples and compatible with nanoscale architectures.

Proteins are the basic building blocks within biological systems and a critical differentiating factor in pathogen identity and function. This project will develop the requisite materials and knowledge necessary to explore and demonstrate, if feasible, the concept of electrochemically controlling the binding of protein at surfaces. Such materials and knowledge will allow for the fabrication of switchable surfaces and their integration into microfluidic, multifunction biosensing devices.

We selected a simple inclusion complex system as the candidate class for exploring this electrochemical switching concept. This system involves the complexation of ferrocene by beta-cyclodextrin, a cyclic polysaccharide. Ferrocene is a stable, electrochemically active species with a standard reduction potential well within the electrochemical potential limits of the aqueous biological realm.

Our previous two years of work demonstrated that beta-cyclodextrin can be functionalized and chemically attached to gold surfaces. Bioconjugate molecules linking biotin with ferrocene were readily synthesized and shown to exhibit appreciable formation constants with the surface bound beta-cyclodextrin. We used the biotin end of this bioconjugate molecule to bind to streptavidin, a protein complex that possesses multiple biotin binding sites.

Specific protein receptors, such as antibodies like immunoglobulin G, can be functionalized with biotin and were shown to be bound to this streptavidin "glue layer." Sequential exposure of beta-cyclodextrin to the sequence of the bioconjugate-streptavidin-antibody creates a surface that is now selective to one type of antigen. This entire sandwich complex can be driven from the surface by oxidation of the ferrocene moiety to the ferrocenium ion and subsequent solvation.

In FY 2006 we focused on improving the stability of this sandwich complex, directly observing and quantifying the electrochemical switching function, and gaining an understanding of the reliability of repeated switching of this structure. We improved complex stability by designing and synthesizing a new ferrocene-based bioconjugate molecule. We developed techniques to directly observe and quantify the switching function by combining a highly sensitive voltammetric technique with nanogravimetry, molecular fluorescence, and plasmon resonance reflectometry.

The combination of voltammetry and fluorescence is particularly noteworthy as these techniques are complimentary to microscale measurements and applicable to microfluidic systems. We demonstrated multiple switching cycles with only moderate loss in subsequent capacity for protein binding. The primary cause of capacity loss appears to be nonspecific binding of residual protein during or after the switching event. We also investigated successful chemical strategies for regenerating the beta-cyclodextrin film.

FY 2006 Accomplishments

We created a set of materials that forms a stable, surface-attached inclusion complex. The inclusion complex is formed from a guest molecule (ferrocene attached to biotin) associating with an electrode surface-bound host (beta-cyclodextrin). Our innovative concept is the synthesis of a first-generation dendrimer – a symmetrically branched molecule – that contains three ferrocene moieties linked to a common biotin group.

We used nanogravimetry and electrochemistry to confirm a greater than five-fold improvement in the retention time of this guest molecule on host-modified electrode surfaces when compared to our original ferrocene bioconjugate. This accomplishment concludes our development of a robust materials set capable of tailoring an electrode for the specific binding of a protein by a simple replacement of the receptor protein.

We demonstrated controlled electrochemical switching by integrating our materials set into a variety of electrochemical, nanogravimetry, and optical platforms. We combined electrochemical techniques with gravimetry, optical fluorescence, and optical plasmon resonance to quantitatively study the switching event. We integrated voltammetry and nanogravimetry into a single platform to study, quantify, and demonstrate this process of switching. We learned that the entire sandwich structure could be released from an electrode under nonflowing conditions with 92 percent dissociating occurring in less than 30 seconds.

We demonstrated the integration and operation of this materials set in microfluidic platforms. We deposited host films onto individually addressable microband electrode assemblies. Preliminary results indicate a correlation between decreases in fluorescence intensity and voltammetrically measured ferrocene loss from the microelectrodes. We used surface plasmon resonance reflectometry to study sandwich complex dissociation in a microfluidic environment using a 10 microliter flow channel at 30 microliter/minute flow rates. Addition of a ferrous/ferric ion solution to the flow stream raised the potential of the electrode to a value greater than 100 mV above the equilibrium reduction potential for the ferrocene dendrimer and triggered fractional loss (40 percent) of the complex.

We tested the stability of this materials set and its degree of reproducible use. Using electrochemical atomic force microscopy (AFM), nanogravimetry, and electrochemical impedance spectroscopy, we learned that overall film structure is maintained. The most significant detrimental effect observed was the nonspecific adsorption of protein onto the film surface after potential-induced desorption. Nonspecific interactions are not mediated by the formation of an inclusion complex and are therefore not controlled and are undesirable events.

Electrochemical AFM revealed aggregate formation of protein. Impedance measurements showed an average increase in the resistance for electron transfer of the film, indicating protein readsorption at the surface. This readsorption process represents a partial loss of function of the film for subsequent specific adsorption of the sandwich complex. Further studies revealed that the capacity of the host film can be regenerated using chemical methods for partially denaturing the nonspecifically adsorbed protein and removing it through solvation. Our studies demonstrate that the host beta-cyclodextrin film is sufficiently stable to endure multicycle electrochemical switching operations.

Significance

The materials created, knowledge gained, and concepts demonstrated within this project will be readily integrated into other existing Sandia projects. A specific example of integration involves the planned incorporation of this materials set into a microfluidic platform designed to explore protein-based cellular functions. One such function where biological sensors are required for direct observation of protein production by a cell is the signaling cascade where an extracellular environmental change induces a cell to respond in some chemical fashion. Cell lysing and protein separation and analysis at timed stages in the cascade allow for mapping of which proteins are responsible for which functions.

The materials set will be integrated into an ultra-capacitor-based sensor. We envision arrays of microcapacitors functionalized with specific receptors as one of several protein detection schemes for this cell signaling work. This work lays the foundation for the successful integration of this materials set and the electrochemical switching concept into such a microfluidic sensing platform for biological system response detection.

Aspects of the general knowledge gained within this project are currently being used in several projects at Sandia. A central theme in nanotechnology is the use of physical and chemical forces intrinsic to a given environment to guide the self-assembly of unique architectures capable of performing specific functions. In a leveraged activity, we are using our growing knowledge of self-assembly to attach porphyrin-based molecules, referred to as nanotweezers, onto self-assembled monolayers based on coordinative interactions between the monolayer film and the binding porphyrin. The nanotweezer is a structurally constrained macrocyclic molecule that is coordinated to a metal cation at its center.

Electrochemical oxidation or reduction of the metal center results in a molecular shape change. An ability to control shape changes at an electrode surface allows for direct electrical to mechanical work conversion at nanoscale dimensions. Strategies exist for tailoring the length scale of this shape change using distance amplification. Assemblies of this type of architecture are envisioned to control nanoscale processes such as reversible, convective flow through nanofluidic channels.

The construction of stable architectures to perform useful work requires both the knowledge of how to guide self-assembly and the tools necessary to determine both final structure and function. The determination of structure and function has been a demonstrated focus of this work.

Refereed Communications

M.J. Farrow, K.R. Zavadil, G.E. Thayer, and B.C. Bunker, “Electrochemically Switchable Beta-Cyclodextrin SAMs for Use in Biomicrofluidic Devices,” presented at the American Chemical Society National Meeting, San Diego, CA, December 2005.

D.L. Pile, K.R. Zavadil, M.J. Farrow, and B.C. Bunker, “Electrochemical Control of Guest-Host Interactions of Derivitized Redox Molecule at Self-Assembled Monolayer Film for Microfluidic Biosensor Applications,” presented at the 209th Meeting of the Electrochemical Society Meeting, Denver, CO, May 2006.

K.R. Zavadil, M.J. Farrow, D.L. Pile, and B.C. Bunker, “Electrochemically Switchable Surfaces Based on Beta-Cyclodextrin SAMS for Biomicrofluidic Applications,” presented at 208th Meeting of the Electrochemical Society, Los Angeles, CA, October 2005.

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Modeling of Friction-Induced Deformation and Microstructure

67075
S. V. Prasad, J. R. Michael, C. C. Battaile, D. J. Bammann, P. M. Gullett, N. R. Moody, P. G. Kotula, E. B. Marin, R. E. Jones

Project Purpose

In FY 2006 we finalized the characterization, interpretation, modeling, and understanding of wear-induced frictional transitions on single crystal nickel (Ni) surfaces. The successful completion of these goals enabled the capability for informed engineering of in-service wear performance of contacting metals in a variety of important applications.

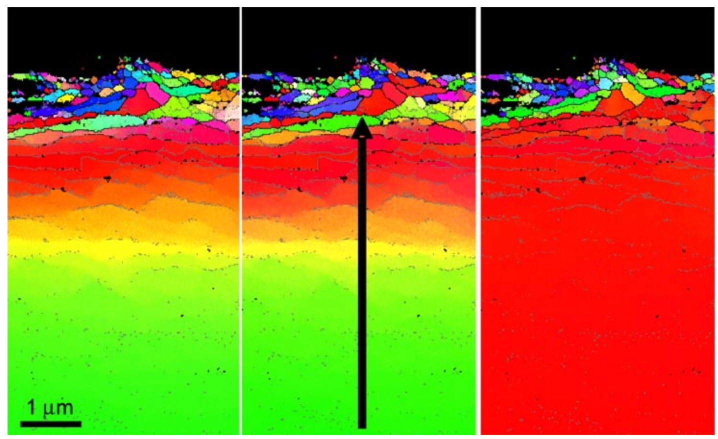
FY 2006 Accomplishments

We completed friction measurements and electron backscatter diffraction (EBSD) characterization of wear-induced subsurface microstructures on single crystal Ni surfaces in various crystallographic directions. Friction data revealed clear transitions from high friction ($\mu \sim 0.8$) to low friction ($\mu \sim 0.2$) around 400 - 600 cycles of sliding, suggesting the possibility of two different friction mechanisms.

Orientation mapping using EBSD provides an excellent means of visualizing the subsurface deformation associated with sliding wear. This work verified that each cycle of wear causes small subsurface orientation changes that eventually result in recrystallization of the surface. Once the surface has recrystallized, the new grains rotate continually with respect to the sliding direction with continued wear cycles to accommodate the further accumulation of plastic strain through grain boundary rotation.

Grain boundary rotation and sliding during frictional contact is verified as one of the important mechanisms of strain accommodation and does result in a decrease in friction coefficient, as has been shown through experimental measurements of friction and as suggested by computer simulation and modeling. This work gave insights to guide the initial simulation work and also provided experimental validation of modeling grain boundary effects on friction.

The insights gained from experiments guided the formulation of a wear model that includes friction, plastic deformation, microstructure formation, and the subsequent modification of the friction coefficient. The loading conditions and material properties parameterize the evolution of plastic deformation, which determines the evolution of microstructure via dislocation subgrain formation, which in turn leads to high ductility in the grain boundary regions and low friction at very small subgrain sizes. Qualitative agreement with experiment is very encouraging.



EBSD provides visualization of subsurface deformation associated with sliding contact on single crystal Ni substrates.

Tribological coatings are often necessary to mitigate friction in electroplated Ni alloys that are of interest to LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) microsystems. The work on single crystals revealed that friction can induce plastic deformation in metallic substrates. We performed a finite element analysis to simulate the extent of friction-induced plastic deformation underneath a diamond-like carbon (DLC) coating in Ni alloys. Experimental friction measurements on coated samples are in agreement with finite element simulations in predicting the load-carrying capacity of DLC film on Ni alloys.

We developed a nanomechanics-based approach to characterize the mechanical behavior of wear surfaces.

We applied this approach, which employs nanoscratch to generate wear patterns and nanoindentation to measure the mechanical properties of wear patterns, to determine the work hardening coefficients in Ni alloys that are of interest to LIGA microsystems.

Significance

Many electromechanical systems that are important to Sandia's core missions contain moving mechanical assemblies (e.g., future generation of stronglinks, sliding electrical contacts) that are subject to friction and wear. In many cases, surface coatings may be necessary to mitigate friction and wear. A clear understanding of subsurface microstructural changes is needed to predict the reliability of moving mechanical systems across all length scales.

In this project, we developed fundamental understanding and modeling capabilities to determine, for specific material and tribological conditions, the nature of the wear behavior of a metal surface. In addition, our modeling work is applicable to larger-scale manufacturing processes. In particular, predicting the friction-induced deformation, self-heating, dislocation cell structure, and recrystallization within a local band at the interface is useful in modeling inertia welding.

Other Communications

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N.R. Moody, M.J. Cordill, J.M. Jungk, M.S. Kennedy, S.V. Prasad, D.F. Bahr, and W.W. Gerberich, "Nanoscale Wear Testing of Single and Polycrystalline Nickel," presented at the Materials Research Society Fall Meeting, Boston, MA, November 2005.

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Reversible Antibody Trapping for Selective Sensor Devices

67076
D. L. Huber, G. D. Bachand, R. P. Manginell, S. M. Brozik

Project Purpose

For homeland security, Sandia needs to develop compact sensors that can selectively detect low concentrations of a wide range of potential biological agents. The most sensitive and selective materials for binding and detecting bioagents are antibodies. Many sensor technologies rely on tethered antibodies for interacting with biospecies. Unfortunately, each antibody is selective to only one antigen. As antibody-antigen complexes form, active sites are consumed, limiting use to one analysis cycle.

The purpose of this project is to study concepts for producing antibody monolayers that can be regenerated and reused. The baseline concept involves using Sandia's "reversible protein trap" to adsorb and release highly selective antibody monolayers. The protein trap consists of a microhotplate onto which a thermally activated polymer (PNIPAM) film is tethered. The hotplate is used to switch PNIPAM between a room-temperature phase that repels proteins and a higher-temperature phase that adsorbs proteins (in this case, antibodies).

The scientific component of this work involves using surface-sensitive Fourier transform infrared spectroscopy (FTIR), interfacial force microscopy, and neutron reflectometry measurements (at the Los Alamos Neutron Science Center) to determine:

- the extent and kinetics of antibody adsorption on activated PNIPAM vs. solution conditions (e.g., competition with other proteins)
- surface concentrations and orientations of the antibodies
- antibody-antigen interactions vs. monolayer structure
- the extent and kinetics of release of antibody-antigen complexes from deactivated PNIPAM films, allowing the surface to be refreshed with the same or a different antibody.

The technical component of the work involves integrating the reversible protein trap into Sandia's highly mass-sensitive shear horizontal surface acoustic wave (SAW) sensors to make a compact device in which selective capture, sensing, and release functions are all performed in the same location. This investigation of concepts for using switchable, "nonselective" surfaces to adsorb monolayers of highly selective agents will impact a wide range of evolving on-chip separation and sensor systems.

FY 2006 Accomplishments

- We confirmed the long-term viability of coatings in real-world conditions. Films tested for weeks showed no signs of degradation.
- We discovered an unusual denaturation process that required the presence of a PNIPAM film, antibody, and antigen to occur. This denaturation occurs at a lower temperature than is observed in any of the constituent proteins.
- We designed a practical chip to integrate heating functionality with the mass-sensing capabilities of SAW devices.
- We confirmed the ability of IgG to resist displacement by competing proteins to maintain its presence and activity in mixed systems.
- We conducted detailed analyses of polymer films, antibody films, and antigen layers using neutron reflectometry, atomic force microscopy, and ellipsometry.

Significance

We developed a unique, reusable sensor system that can be easily integrated into a versatile biosensor. There has been substantial interest from microsystems researchers to continue development of this system for fieldable homeland security systems.

The polymer syntheses perfected in this project have great versatility and can be applied to form nanoscopic polymer films of almost any polymer to almost any substrate. These films are of excellent quality and are free of pinhole defects. The versatility of the chemistry will allow this simple procedure to be applied to numerous surface functionalizations.

Other Communications

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Correlated and Comprehensive Analytical Techniques for Homeland Defense

67077

P. G. Kotula, D. K. Melgaard, J. A. Ohlhausen, D. E. Peebles, B. L. Doyle, M. R. Keenan

Project Purpose

The purpose of this project is to:

- better characterize and perform comprehensive bioforensic analysis of potential bioterror materials
- correlate several different analytical imaging (full spectral imaging) techniques to learn what new/existing individual analytical imaging techniques can tell us about methods of production/chemical signatures
- rigorously correlate multiple analytical imaging techniques with new/existing multivariate statistical analysis algorithms/tools
- perform correlated materials analyses on both test structures as well as simulant biothreat materials to test the validity of the new approaches.

FY 2006 Accomplishments

Algorithm Development/Implementation

- Implemented full 64-bit version of our automated eXpert spectral image analysis (AXSIA) software on our dual itanium 64-bit computer with 6Gb random-access memory
- Implemented double-precision version of AXSIA for high-dynamic-range data and correlated data analysis
- Demonstrated the ability to perform one single analysis on a series of individual spectral images within AXSIA
- Implemented equality constraints within AXSIA whereby an analysis can be seeded with a known spectral shape(s) to reduce the effect of nonzero background intensity or account for the effect of instrumental instability such as energy-axis shift/drift in x-ray photoelectron spectroscopy or electron energy-loss spectroscopy
- Developed a new algorithm called Principal Factor Analysis that requires no a priori knowledge of the uncertainty in the measurements or noise structure and has higher sensitivity to minor chemical features in the data than previous alternating least-squares-based methods.

Spectral Image Correlation/Analysis

- Correlated data from the same instrument for different specimens, same specimen under different acquisition conditions (detector/specimen orientation, different accelerating voltages and/or magnifications, and/or rotations)
- Correlated data from different depths within the same specimen (tomographic spectral images)
- Correlated data from focused probe (scanning transmission electron microscopy) system consisting of simultaneously acquired x-ray and electron energy-loss spectroscopy data
- Correlated data from two different instruments, scanning electron microscope x-ray spectral images and time-of-flight secondary-ion mass spectrometry
- Concatenated data, adjusted it for alignment and distortions, and analyzed it with AXSIA.

Significance

This work is widely applicable. Our new capabilities will have direct bearing on and application to our current and future nuclear weapons work where we often are faced with finding minor chemical signatures due to aging and/or manufacturing/remanufacturing activities. This work allows us to expand beyond single independent analytical techniques into the realm of correlating two or more analytical techniques. This work is already being applied to and leveraged by other current projects, including one for the Department of Homeland Security.

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Development of High Energy Density Dielectric Materials for Integrated Microsystems

67078

B. A. Tuttle, G. A. Samara, E. L. Venturini, J. Cesarano III, M. A. Rodríguez, D. A. Hoke, J. A. Voigt, V. K. De Marquis, P. G. Clem

Project Purpose

Next-generation surety systems will require compact, highly integrated microsystems to improve device functionality, miniaturization, and performance. The purpose of our work was to investigate and develop dielectric materials with the largest energy storage densities possible. These materials permit reduction of the size of what is often the largest component of a microsystem: the capacitor. Since capacitors are often the largest components in these systems, the greatest increase in volumetric device efficiency can be gained by reducing capacitor size.

In the last several years, dramatic developments in dielectric science have been reported that can enable 2X to 100X reduction in capacitor size over state-of-the-art commercial materials. We have investigated two new dielectric material families as alternatives to state-of-the-art $\varepsilon_r \sim 1100$ dielectrics: 1) specially formulated lead lanthanum zirconate titanate (PLZT) dielectrics, and 2) novel relaxor dielectrics such as $CaCuTiO_3$ (CCTO).

A further purpose of our project was to develop materials processing techniques that permit integration of these complex materials into integrated microsystems. Thus, our overall goal was to develop new materials for high-field applications and integrate them into prototype substrates that are compatible with both Sandia and commercial microsystems using direct-write technologies.

FY 2006 Accomplishments

We developed advanced processing techniques for the direct-write processing of PLZT thick films using aerosol spray deposition. An important development was the incorporation of lead germanium oxide with chem-prep PLZT powders using sol-gel methods. This permitted fabrication of high-quality thick film PLZT layers at temperatures of 950 °C directly onto alumina substrates.
We performed pulse discharge tests on Sandia-integrated PLZT dielectrics. The results of these tests indicated that these capacitors could withstand over 100 discharge cycles at high voltage. We developed advanced processing techniques of fabricating multi-layer capacitors consisting of six active dielectric layers deposited directly on alumina by aerosol spray deposition.

Perhaps our most important technical accomplishment was completing the comprehensive study of pressure and temperature phase relationships of high zirconia content PLZT ceramic materials. We also achieved extremely significant CCTO accomplishments. These included the process developments leading to the first-ever fabrication of CCTO thin films (less than 2 micrometers thick).

An equally important accomplishment was the development of procedures for thick film capacitors based on CCTO dielectrics directly onto alumina. We developed an in-depth scientific understanding of the mechanism for the novel, high-impact CCTO dielectric response of extremely high dielectric constants that are temperature insensitive over large temperature ranges.

Significance

The expected decrease in capacitor size by a factor of two is significant for both commercial and Sandia applications. (Sandia component engineers stated that they would implement capacitors with only a 30 percent reduction in size into their systems immediately.) Further, the Sandia reduction in the processing temperatures of high zirconia-content PLZT dielectrics by 250 °C to 950 °C is significant.

Interest at Sandia has been verified by new projects generated from this work. A follow-on Sandia project for multilayer PLZT capacitor fabrication and prototype testing was initiated at the beginning of FY 2006, and a second follow-on project began in late FY 2006.

Our developments also mean that, for commercial applications, more inexpensive electrode materials and packaging technologies can be used. Among the applications that could be impacted are direct-current bus capacitors for inverters for fuel cell and electric hybrid vehicles, and medical electronics applications such as in situ defibrillators for pacemakers. Our discovery and validation of the mechanism of CCTO high-value dielectric response may also lead to low-voltage capacitor devices for cell phones and other commercial integrated microelectronics.

The scientific understanding developed in this study has led to more than 10 invited talks by Sandia scientists at technical meetings throughout the course of this project.

Refereed Communications

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Nanolithography Directed Materials Growth and Self-Assembly

67079

J. W. Hsu, W. M. Clift, D. R. Tallant, M. E. Coltrin, J. A. Voigt, N. C. Simmons

Project Purpose

The purpose of this project is to discover new methods for patterning nanomaterials and to understand the role of surface structure and chemistry on crystal nucleation and growth. The growth/assembly of inorganic nanomaterials has great impact on creating new devices for applications in bio/chemical sensing, optoelectronics, catalysis, and energy conversion. We are taking a "bottom-up" rather than "top-down" approach. Whereas conventional optical and electron-beam lithography function in a "top-down" manner, we instead adapt soft nanolithography techniques to create regions with different surface energy, resulting in selectivity in crystal nucleation and growth morphology.

We successfully demonstrated this approach for the growth of ZnO nanorods on Ag surfaces, including exquisite control in spatial selectivity, crystal orientation, crystal shape, growth morphology, and nucleation density. In principle, this approach is applicable to a wide variety of inorganic crystals and substrates, depending on the desired applications. To optimize the process, understanding the role of surface structure and chemistry in the assembly process is critical. We examined and identified applications that rely on precise control of nanostructure morphology.

FY 2006 Accomplishments

We developed a better understanding of the critical factors that affect ZnO nanorod nucleation and growth on Ag films. In addition, we examined the formation of alkanethiol and alkanedithiol monolayers on GaAs surfaces. Molecular monolayer formation is critically important in surface modifications and organic templating of inorganic crystal growth.

Effect of Patterning on Nucleation Density: Experiment and Modeling

In previous years we found that the density of ZnO nanorods depends on the size of active growth regions as well as the fill factor of the pattern. We grew ZnO nanorods on different patterns and compared the experimental results to a model we developed based on modification of previous work on selective area growth in metal-organic chemical vapor deposition.

Characterization of Nanorod Optical Properties

We performed photoluminescence, photoluminescence excitation, and dynamic studies coupled with sample annealing treatments. We found that these nanorods are oxygen rich as grown and become more stoichiometric when annealed in reducing atmosphere. We performed cathodoluminecese imaging and low-temperature photoluminescence and electron irradiation studies to understand the nature of subbandgap luminescence in the as-grown ZnO nanorods.

Molecular Monolayer Endgroup Functionality Studies

We found that ZnO nanorods grow on bare Ag surfaces but not on the self-assembled monolayer (SAM) regions when methyl terminated alkanethiols are used. Scanning Auger results showed that Zn does not bind to methyl endgroups of SAM as expected, resulting in inhibition of ZnO crystal nucleation. When carboxylic endgroup SAMs are used, we observed two types of behaviors: Type 1 areas have lots of ZnO nanorods, while type 2 areas have none. However, we found Zn signal in both types of areas despite the lack of nanorod growth in type 2. Hence, preferential binding of growth species is not necessary for nanorod growth. We employed cross-sectional transmission electron microscopy to investigate the ZnO on the SAM surfaces.

Ag Crystallographic Orientation Studies

All our work so far uses Ag surfaces made by electron beam evaporation of Ag films on Si substrates. These films exhibit (111) textures, but the grain size is very small (~ 10 nm), and there is no in-plane orientation among different grains. Recently we used Ag foils with proper polishing and annealing treatment as substrates for ZnO growth. Interestingly, we found strong preferential nucleation on (111) oriented grains but not on grains of other orientations. Furthermore, all ZnO nanorods are oriented the same way.

Significance

The ability to control nanostructure morphology has great impact in applications that use these nanomaterials, including chemical/biological sensors, light sources and detection, catalysis, and solar energy conversion. We investigated using ZnO nanostructures for these applications and determined that the most promising application is using ZnO nanostructures as the electron transporting materials in organic-inorganic hybrid solar cells. In these applications, the surface area, spacing, and order of the materials have dramatic impact on the device performance. The critical factor is the spacing between neighboring ZnO nanorods, which should match the exciton diffusion length in the polymer matrix, typically ~ 10 nm.

Our ability to grow ZnO nanostuctures at the nanolength scale led to a new LDRD project that focuses on solar cells, with close collaboration with a team at the National Renewable Energy Laboratory. This work leverages the expertise at Sandia and will produce results relevant to DOE's mission.

Our work on this project work also led to four refereed publications in premier journals, several invited talks at international meetings and universities, and contributed talks and posters.

Refereed Communications

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Development of a Novel Technique to Assess the Vulnerability of Micromechanical System Components to Environmentally Assisted Cracking

67080
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Project Purpose

Microelectromechanical systems (MEMS) will play an important functional role in future DOE weapon and homeland security applications. If these emerging technologies are to be applied successfully, it is imperative that the long-term degradation of the materials of construction be understood.

Unlike electrical devices, MEMS devices have a mechanical aspect to their function. Some components (e.g., springs, contacts, inertial sensors, and so on) will be subjected to stresses beyond whatever residual stresses exist from fabrication. These stresses, combined with possible abnormal exposure environments (e.g., humidity, contamination), introduce a vulnerability to environmentally assisted cracking (EAC).

EAC is manifested as the nucleation and propagation of a stable crack at mechanical loads/stresses far below what would be expected based solely upon the materials' mechanical properties. If not addressed, EAC can lead to sudden, catastrophic failure. Considering the materials of construction and the very small feature size, EAC represents a high-risk environmentally induced degradation mode for MEMS devices.

Currently, the lack of applicable characterization techniques is preventing the needed vulnerability assessment. The objective of this work is to address this deficiency by developing techniques to detect and quantify EAC in MEMS materials and structures under atmospheric conditions relevant to the anticipated service conditions.

In FY 2006, we worked toward several specific goals: further refine and expand the capabilities of the test technique defined in FY2004-2005; pursue a new surface damage detection technique; and establish the threshold humidity level required for the atmospherically controlled EAC of thermally processed Watt's nickel at a fixed gaseous contamination (chlorine) level.

FY 2006 Accomplishments

Our research had three main thrusts:

Refine the sample geometry from plated and machined samples to completely as-plated samples (i.e., a plated-in notch, rather than a machined notch)
The properties of LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) materials are a strong function of their as-plated microstructures. In this case, we made a mask and plated two complete wafers of parts. The test samples consisted of four different notch geometries, ranging from a 125 micron radius down to an approximately 5 micron root radius, the latter simulating a sharp crack. We used the stress concentration factor for a thin, notched bend bar to establish the appropriate load for each sample, based upon the acuity of the notch and the physical properties of the material.

Document the susceptibility of Watts nickel-based LIGA parts to atmospheric environmentally assisted cracking within a chloride-containing environment
We evaluated materials using the semiautomated, controlled atmosphere mechanical test system developed and refined during FY2004-2005. We evaluated samples of each notch geometry at applied loads, which resulted in a similar maximum stress in front of the notch. The exposure environment was air with 50 ppb Cl_2 at 30 °C under a range of humidities. These experiments demonstrated a clear threshold

relative humidity above which the material cracked. This threshold humidity level was independent of the notch geometry for the loads we evaluated.

Evaluate a new test method to observe the early stages of damage/crack initiation optically
This technique is based on differential imaging microscopy and allows the researcher to deconvolute optically complex images (due to surface morphology, and so on), extracting only the degradation response (represented as changes in the image).

Due to physical constraints presented by the existing mechanical test system, the initial material system evaluated was gold-plated copper, similar to that used in microelectronic connectors. The existing test system was too compact to allow for the addition of a highly stable optical train; adding such a technique to the existing system would have required substantial reworking of the environmental chamber, reducing considerably the resources available to actually perform experiments once the system was assembled. We selected gold-plated copper because it dramatically simplified implementation of the technique, samples of an acceptable geometry could readily be acquired, and the reduced sample size required only that a simple environmental chamber be constructed.

We assembled an imaging system consisting of a camera, optical train, and mechanical translation stages. Proof of concept experiments demonstrated that this technique is capable of monitoring subtle changes in the surface, in this case the initiation of pore corrosion, previously unresolved experimentally.

Significance

Several accomplishments of this project will impact future work. The first is that atmospherically controlled environmentally assisted cracking of MEMS materials, in this case LIGA nickel, can indeed occur given the proper combination of material and environment. This is a degradation/failure mechanism that is generally not considered while constructing such devices, but which would be of very high consequence should it take place in the field.

The second is the construction of the optical system for differential imaging analysis, combined with the initial proof of concept work. This technique will have applications in numerous material degradation analyses. It will allow, for example, the direct observation of electronic and microelectronic degradation processes, such as aluminum-gold wirebond corrosion or the atmospheric degradation of precious metal plated materials, along with numerous other degradation phenomena.

Past work in all of these areas has been limited to viewing things after they occur, estimating reaction kinetics, and so on. The differential imaging technique permits these processes to be observed directly, allowing for a more complete and precise understanding of degradation phenomena used within many system lifetime models. The initial success demonstrated in this project has led to a separate research effort to begin in FY 2007 aimed at further developing the differential imaging technique.

3D Optical Sectioning with a New Hyperspectral Deconvolution Fluorescence Imaging System

67081

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Project Purpose

This project included the design, construction, and use of a novel hyperspectral fluorescence microscope for high-resolution three-dimensional (3D) optical sectioning of cells and subcellular organelles, 3D monitoring of microfluidic processes, investigation of molecular motors, and imaging of photosynthetic cyanobacteria.

We developed and extended new data analysis methods to deconvolve the hyperspectral image data and to rapidly extract quantitative 3D concentration distribution maps of all emitting species. The imaging system has many advantages over current confocal imaging systems, including simultaneous monitoring of numerous highly overlapped fluorophores, immunity to autofluorescence or impurity fluorescence, enhanced sensitivity, and dramatically improved accuracy, reliability, and dynamic range. Efficient data compression in the spectral dimension allows personal computers to perform quantitative analysis of hyperspectral images of large size without loss of image quality.

The new imaging system is an enabling technology for numerous applications, including 1) 3D composition mapping analysis of multicomponent processes occurring during host-pathogen interactions, 2) monitoring microfluidic processes, 3) imaging of molecular motors, and 4) understanding photosynthetic processes in wild type and mutant *Synechocystis* cyanobacteria.

FY 2006 Accomplishments

The 3D hyperspectral imaging system is fully operational and obtaining excellent quality hyperspectral images. We carefully characterized the noise characteristics of this diffraction-limited confocal imaging system and developed methods to quantify the statistical significance of each component in each pixel.

Further improvements in the software for operation and visualization of the data have been completed, and multivariate curve resolution (MCR) software for extracting pure emission spectra and relative concentrations has continued to be upgraded with the new formation of a graphical user interface (GUI) program. This GUI-based software not only significantly improves the ease of operation of the software, but also makes it possible for nonexperts to use the software.

Work progressed in two applications (imaging motor proteins and the investigation of lipid bilayers). In addition, we investigated multiple new applications with the new hyperspectral imaging system. For example, we continue to image live *Synechocystis* wild-type and mutant bacteria and have submitted a paper to the *Proceedings of the National Academy of Sciences* on this ground-breaking research.

In collaboration with Professor Diane Lidke of the University of New Mexico Health Sciences, we initiated a task to image single quantum dots in rat basophilic leukemia cells to study cell signaling events in individual cells. We demonstrated that the new microscope can image single quantum dots in the cells and that we can easily and simultaneously separate the five classes of quantum dots present in the cells. We also imaged live bacteria in biofilms and for the first time imaged the bacteria on autofluorescent filter membranes in a study important for improving water supplies and water quality.

Significance

The new hyperspectral confocal fluorescence microscope, along with the MCR software used to analyze the image data sets, is unique and offers a new technology by which biologists can study complex processes occurring in living cells. It allows much more quantitative information to be obtained from the confocal images than possible with current commercial microscopes when autofluorescence and multiple fluorophore labels are present in the cells. Applying the MCR software to the hyperspectral images allows the pure spectra of the fluorescing species to be "discovered" directly from the image data and their relative concentrations to be accurately displayed in 3D without the need for standards or a priori information.

In collaboration with Professor Willem Vermaas at Arizona State University, we are able, for the first time, to accurately and quantitatively image the six spatially and spectrally overlapping photosynthetic pigments in *Synechocystis* wild type and mutant cyanobacteria at diffraction-limited spatial resolutions (250 x 250 x 600 nanometers). These results show that hyperspectral fluorescence imaging can provide unique quantitative information regarding heterogeneity of pigment organization and localization in the cells even when there is high spectral and spatial overlap of the fluorescent pigments.

This challenging project is positioning Sandia as a major player in advanced spectral imaging techniques for materials research and biotechnology. Realization of this new system resulted in submission of external research proposals for homeland security, Genomics:GTL, DOE Basic Energy Sciences, and National Institutes of Health projects. It led to a large cooperative research and development agreement with Monsanto for biofuels applications, led to collaborative research with the University of Texas Medical Branch, serves as the imaging component of a Grand Challenge LDRD project to study host-pathogen interactions, and provides hyperspectral imaging support to three additional Sandia LDRD projects.

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Other Communications

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The Science of Solutes: Transition Metals in LIGA Nickel

67082
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Project Purpose

All engineered materials contain solutes whether intentional (alloying elements) or unintentional (impurities). Because solutes interact with the material microstructure, solute effects extend beyond modifying chemistry to the extent that solutes may control material properties even when they are present in tiny amounts. Although solutes control both the processing and the properties of most alloys, material and process models almost never include their effects. Yet without physically based models for the effects of solutes, the interpretation of experimental data has been heuristic at best and anecdotal at worst.

In this project, we made the first concerted effort to include realistic solute effects in models for technologically important materials. Nickel-manganese (Ni-Mn) electrodeposits are attractive candidates for a variety of microsystems applications that require high strength and ductility, such as stampers for large-area nanopatterning and high-density storage media.

While electroforming is far more adaptable to microfabrication than, for example, casting, the metallurgy of electrodeposits is considerably more complicated than that associated with the more traditional means of producing metal parts. This is because the alloying elements in electrodeposits can affect both the deposition process as well as the properties of the finished product. For example, small amounts of transition metal (TM) dopants such as Mn are incorporated in LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) Ni in order to refine the as-deposited grain size in the absence of sulfur-bearing additives.

However, the consequences of Mn additions extend beyond grain size refinement. Mn changes deposition texture; it stabilizes the as-deposited grain structure by inhibiting recrystallization and grain growth, and it appears to strengthen the material beyond what would be expected based on grain refinement alone. Preliminary evidence indicates that other TMs may have similar or even greater influence on the properties of Ni electrodeposits.

Discovering how TMs accomplish these changes and optimize composition to maximize them are the goals of our coupled experimental and computational efforts. Our approach is to characterize a representative set of Ni/TM samples, which vary in process history (deposition conditions, composition, and so on), using techniques ranging from the atomic (atom probe microscopy) through the mesoscale (transmission electron microscopy (TEM), scanning electron microscopy) to the continuum (x-ray diffraction (XRD)). Computational models at all three scales used experimental results and inform experimental efforts. Using this combined approach, we optimized the material by understanding the science.

FY 2006 Accomplishments

In this project, we performed extensive experimental examination of the thermal stability of Ni-Mn electrodeposits. In parallel, we used computational modeling to examine proposed stability mechanisms. Specifically, we provided accurate and detailed description of the annealing response of nanocrystalline Ni and Ni-Mn electrodeposits over a wide range of length scales, with the ultimate goal of understanding the role of Mn impurity in thermal stabilization of Ni.

Using a combination of mechanical testing, XRD, TEM, and three-dimensional (3D) atom probe tomography, we established how yield strength, preferred grain orientation (texture), average grain diameter, and impurity distribution on atomic scale change in Ni and Ni-Mn alloys. We also established the capability to fabricate and analyze Ni-Fe electrodeposits to compare with Ni-Mn.

We developed a twinning model that takes a 3D grain structure and forms twins in a physically and crystallographically realistic manner and analyzed our twinned structures via polycrystal plasticity finite element modeling, determining that twinning does not significantly affect stress-strain response.

We also explored the influence of transient solute concentration gradients on microstructural stability using a mesoscale cellular automaton, observing a newanomalous growth mechanism. Finally, we used molecular dynamics to perform the first 3D measurements of grain boundary stiffness, discovering an unexpectedly large variation of the stiffness with direction.

Significance

The results of this project are significant to DOE, the National Nuclear Security Administration, and the worldwide scientific and technical community interested in metallurgy. Specifically, Ni electrodeposits continue to be used in variety of defense program-related work, and understanding, controlling, and predicting the properties of such components is essential to successful management of the stockpile. Furthermore, the scientific achievement of this project have direct relevance to the rapidly growing and expanding optical data storage technology, where there is a growing need for strong, and thermally stable Ni based alloys for nanoembossing.

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Novel Gel-Based Technology for Sensors and Weapons

67083

J. L. Lenhart, P. J. Cole

Project Purpose

The purpose of this project is to identify and understand the physical and chemical factors that control the mechanical and adhesive properties of soft polymer gels and to use this information to design gel materials for various applications at Sandia.

A gel is a physically or chemically cross-linked polymer that is highly swollen by solvent. Mechanically, the solvent creates a “soft solid,” which is easily deformable yet still recovers from deformation due to the elastic nature of the cross-links in the polymer. Polymer gels offer potential in a wide array of applications, because the gel properties can be tuned by varying the polymer type, solvent type, and solvent loading. In addition, small molecule additives and fillers can be incorporated into the gel formulation to further enhance the properties. Potential applications include drug delivery, biomedical implants, artificial muscles, food and cosmetics, separation systems, display devices, electronics, batteries, optical devices, and sensors to name a few.

While varying solvent type and loading provides a wealth of potential applications, the high solvent loading invokes unique materials challenges, as the solvent-polymer partitioning impacts the gel microstructure and the resulting properties. Solvent-polymer partitioning in a gel can be critical for the performance of devices. For example, polymer gels can undergo a volume phase transition when external conditions such as temperature, pH, solvent, or concentration of chemical or biological analytes is altered.

In some applications, this swelling-shrinking phenomenon can potentially be exploited to make devices such as are sensors for biological and chemical contaminants, controlled lubrication layers, and so on. In other applications, it is undesirable to have a gel exhibit phase separation, particularly when gel adhesion to a substrate is critical for device performance.

The objective of current research is to design a polymer gel that will perform over very broad temperature ranges both above and below room temperature. Key to this objective is a fundamental understanding regarding the role of the polymer-solvent interactions on the gel phase behavior, partitioning, microstructure, and properties.

In FY 2004 and early FY 2005, we designed silicone-based gels for implementation in weapons devices. In FY 2006 we focused on polybutadiene- and epoxy-based gels for sensor applications. We used a combination of rheology, adhesion, and scattering measurements to probe the gel and make critical links between microstructure and properties.

FY 2006 Accomplishments

The major accomplishments of FY 2006 are 1) understanding the links between solvent loading and the resulting polymer microstructure, 2) investigating the impact of polymer cross-link density on the gel mechanical and adhesive properties, 3) identifying whether subtle chemical changes can invoke a thermodynamic response in the polymer gel, and 4) investigating the potential to exploit gel technology for sensor applications.

Accomplishment 1

The solvent loading had a profound impact on the polymer microstructure. In particular, addition of solvent during polymer cure resulted in a lower entanglement density relative to a polymer cured initially without solvent and then swollen to the same solvent content. Interestingly, for lightly cross-linked gels, the first-order effect on the gel modulus was the level of entanglements. A lesser second-order effect was actually the solvent loading.

Accomplishment 2

Polymer cross-link density had a dramatic impact on the gel adhesive and mechanical properties. In particular, tack adhesion for the solvent swollen gel behaved similarly to that of elastomeric adhesives in that the tack increased with decreasing cross-link density. The tack adhesion also increased with decreasing temperature as the glass transition temperature of the gel was approached due to visco-elastic dissipation in the gel at low temperatures. However, the tack adhesion was impacted by solvent exclusion processes that were temperature-dependent.

The cross-link density of the polymer could be used to control the glass transition temperature and plateau shear modulus of the gel formulations. The cross-link density had a substantial impact on the polymer microstructure with solvent addition, as the modulus reduction with increasing solvent loadings was more pronounced in lower cross-link density polymers.

Accomplishment 3
We developed epoxy gels where subtle chemical changes were incorporated by attaching alkane chains to a small fraction of the reactive groups. Modifying the gel in this manner did result in small changes in equilibrium swelling of the polymer. However, the swelling changes were not catastrophic, suggesting that nonaqueous gels are not directly useful in sensor applications

Accomplishment 4
Gel technology can be used in sensor applications if the solvent is exploited as a processing aid to control the gel microstructure. In particular, after removing the solvent, high-porosity polymeric scaffolds can be developed as a sensor platform.

Significance
Initially, we focused our efforts on applications critical to the weapons mission of Sandia, as polymer gels were being proposed for various devices. The knowledge developed regarding gel adhesion and the impact of solvent loading was important for designing materials that could meet stringent device requirements. More recent research focused on exploiting gel technology for alternative applications such as sensors for chemical and biological agents. One of the critical obstacles to subtle chemical detection is obtaining adequate signal in trace chemical environments. Gel technology can be used to develop high-porosity and high-surface-area polymeric scaffolds that serve to concentrate the chemical environment and potentially lead to high signal. This idea is being pursued, and follow-on funding is anticipated to develop practical sensor devices exploiting this porous polymer technology.

Refereed Communications
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Other Communications
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J.L. Lenhart and P.J. Cole, “Soft Polymeric Materials: Fundamental Understanding to Practical Application,” presented (invited) at University of Louisville Department of Chemical Engineering, Louisville, KY, April 2006.

Coupled Nanomechanical Oscillator Arrays for the Study of Internal Dissipation in Nanoscale Structures and Collective Behavior in Large Systems

67084

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Project Purpose

Controlling energy dissipation in nanostructured materials is a fundamental materials issue for Sandia's future micro- and nanosystems. The goal of this project is to understand internal dissipation in structures of nanoscale dimensions. In a new approach, we studied large arrays of coupled nano-mechanical oscillators combined with theoretical calculations aimed at identifying atomistic mechanisms of dissipation. This research would be especially relevant to the nanomechanics thrust area of the Center for Integrated Nanotechnologies and to the development of nanomechanical resonators for homeland security applications (e.g., sensors).

We made measurements at low temperatures, where dissipation due to tunneling states and ballistic phonon transport become important, and developed a new theoretical approach combining large-scale classical force field techniques with first-principles quantum mechanical calculations to identify the dissipative defects, such as the ubiquitous tunneling states defects in amorphous materials. The large system of coupled nanomechanical elements provides a unique experimental platform for understanding major problems in solid-state and statistical physics.

Coupled oscillator arrays, which resemble two-dimensional (2D) ball-and-spring models, can be used to study disorder-induced localization in 2D systems and stochastic resonance and nonlinear effects in coupled systems. Specifically, we examined localization of vibrational modes of the array at intentionally introduced defects and coupling of these modes to mechanical noise leading to mode amplification (stochastic resonance) or the emergence of complex phenomena in the case of bistable oscillator arrays (which are a mechanical analog to biological neural networks).

FY 2006 Accomplishments

We made significant accomplishments in three primary areas this year:

1. We fabricated 2D arrays and one-dimensional (1D) linear chains of coupled oscillators in both amorphous and crystalline diamond materials, extending coupled oscillator physics studies into new geometries and new materials systems.
2. We completed defect-related dissipation studies in materials systems ranging from amorphous to crystalline diamond, revealing new defect-related dissipation processes.
3. Our theoretical studies of defect-related dissipation showed that a single isolated defect in an otherwise perfect crystal can strongly impact internal dissipation.

The coupled oscillators consisted of arrays of 100 x 100 oscillator elements, with the oscillator defined as a node in a grid fabricated from amorphous diamond-like carbon having a tungsten proof mass on top. These oscillator arrays exhibit vibrational modes similar to drumhead modes of a stretched membrane. Resonance testing of the arrays revealed a series of resonance peaks, where the lowest frequency peaks were in reasonable agreement with a modal analysis of the simulated array using finite element modeling. The 1D arrays were fabricated out of polycrystalline diamond and consisted of a chain of cantilever oscillators with e-beam lithographically defined thicknesses down to 100 nm and widths of 700 nm. These oscillators have their dominant resonance mode in the plane of the film.

Our primary testing on these arrays was to identify the defect-related mechanical dissipation mechanism. We observed defect-related mechanical dissipation in this material, similar to what we found earlier in amorphous diamond-like carbon films, but the nature of the defect-related dissipation process was very different. In particular, much lower dissipation is observed in crystalline diamond films, with Qs exceeding 100,000 in contrast to the typical Q of 4000 observed for amorphous films.

Furthermore, the defect that gives rise to dissipation in boron-doped diamond films is absent in the undoped films. This boron-related defect gives rise to a peak in the mechanical dissipation at about 673 K. No peak in dissipation was observed in amorphous carbon or undoped diamond films. The atomic nature of the defect that controls internal dissipation remains to be identified.

One avenue to identifying the atomic nature of the defects is to perform computer simulations of atomic defect-related dissipation. We performed a study of internal dissipation in crystalline silicon containing a single split interstitial defect. Our work showed that the presence of an isolated defect leads to a significant reduction in Q (about 25 percent) even at the frequencies where the defect relaxation time constant is not matched to the oscillator period. Stronger dissipation is expected when these time constants are matched, and our estimates suggest this should occur at about 500 K.

This work provides the first clear ideas of how a single isolated atomic defect gives rise to mechanical dissipation in a larger-scale oscillator.

Significance

Micro- to nanoscale mechanical oscillators are the keystone to many breakthroughs in nanoscience, such as single electron spin detection using magnetic resonance force microscopy or nanoscale displacement measurement using a nanoscale oscillator coupled to a single electron transistor. It is critical that these micro- and nanoscale oscillators exhibit low internal dissipation in order to provide useful function. Unfortunately, our understanding of internal dissipation in small mechanical structures is limited.

The objective of this project is to determine the fundamental mechanisms of energy dissipation in nanoscale structures through experiments on coupled nanoscale oscillators and theoretical calculations of atomic-scale dissipation mechanisms. This research is leading to an understanding of dissipation mechanisms in nanoscale structures and materials having internal structure at the nanometer or less length scale (i.e., nanostructured and amorphous materials).

Our work in this project showed that atomic scale defect relaxation processes can dominate mechanical dissipation even in nanoscale oscillators. We identified the activation energies for defect relaxation processes in amorphous and crystalline diamond oscillators and developed experimental approaches that are applicable to a wide range of oscillator materials. By identifying the mechanism of internal dissipation and the activation energies for the defect relaxation process, it is now possible to predict the dependence of internal dissipation on temperature and to guide the selection of material for high-Q oscillators.

For example, we found that boron-doped diamond films exhibit significantly higher dissipation than undoped films (up to a factor of three), suggesting that undoped diamond films are more suitable for high-Q oscillators, and noncapacitive approaches should be considered for this material (i.e., optical detection).

Our theoretical studies of atomic-defect related dissipation offer great promise for the fundamental study of defect-related dissipation. This new approach should enable understanding of how a single defect gives rise to mechanical dissipation and provides an opportunity to use mechanical dissipation studies as a tool for defect spectroscopy in a wide class of materials.

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N. Sepulveda, D.M. Aslam, and J.P. Sullivan, "Fabrication and Testing of Polycrystalline Diamond Nanoresonators," presented at the 2006 MRS Spring Meeting, San Francisco, CA, April 2006.

Other Communications

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Precisely Controlled Picoliter Vessels with Rapid Sample Preparation for Trace Biotoxin Detection

67085

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Project Purpose

The purpose of this project was to explore the synthesis and characterization of novel microstructures for discrete, controllable sample management in microsystems. This effort demonstrated a practical and simple solution for handling picoliter biomimetic volumes on-chip by synthesizing robust vesicles and applying electrically monitored electroporation.

As containers, vesicles are made of an engineerable wall material capable of being selectively permeated, which when sealed, prevent samples from escaping. We advanced the field of vesicle materials science by developing large, robust, synthetic analogues of natural vesicles to enable control over microsamples and microreactions in ways not previously attainable.

As substrates, vesicles are ideal for low-reagent volumes and fast surface-dominated reactions required in the preparation of any assay. We envision a device in which analytes are trapped in vesicles when they come in contact with a prefabricated lipid film on a chip, moved through a chip without broadening to form various combinations with other encapsulated analytes, and then simultaneously released to initiate reaction, separation, or detection.

There are many applications for such a system: in biochemistry as a platform for study of molecular dynamics, in chemical/biological threat detection as a dilution-free conveyance and preparation motif, and in medicine as an accurate and local drug delivery technique.

FY 2006 Accomplishments

We developed reliable and convenient ways to make and use vesicles in a microfluidic context and showed that these vesicles can be successfully trapped and electroporated multiple times. This was the first demonstration of controlled vesicle electroporation in an on-chip microfluidic platform. We made significant progress in each of the three major areas of investigation: vesicle formation, vesicle behavior during electroporation, and design of a multitrap microfluidic device for vesicle handling.

We developed a new method for preparing vesicles that is compatible with field applications in a "just add water" mode, in which dried vesicles on a stored microfluidic chip are rehydrated immediately before use. Effective vesicle preparation methods have previously been reported using evaporation of a nonaqueous lipid solution, film hydration, and dispersal in an aqueous buffer. However, these schemes are not suitable for most microfluidic applications due to problems with reproducibility, polydispersity, and vesicle mechanical strength.

By adding materials directly to the lipid solution and adjusting buffer composition, we modulated the osmotic pressure in vesicles, improving their mechanical strength and gaining greater control over size and shape. We can also introduce functional materials such as fluorescent dyes by this approach. Our procedures create far less hazardous waste than is reported for earlier procedures.

We also invented an approach in which a gel is formed in vesicles shortly after they are created. The gel is analogous to the cytoskeleton commonly found in cells. Microfluidic mechanical testing of these vesicles shows that, in the absence of gel, vesicles are difficult to maintain in a trapped state. On the other hand, the reinforced vesicles exhibit a wide window of pressures under which they can be trapped and manipulated. This improvement is likely to be an essential feature of practical applications of vesicles as microfluidic cargo containers.

We created a new microfabricated polydimethylsiloxane single-cell electroporation device this year. The device is unique in that it contains sequential/multiple trapping locations that minimize the mechanical stress on the vesicles. This opened a new application space for single-cell analysis. Previously published devices are not suitable for long-term studies since they induce mechanical deformation on cells, which leads to cell death. We verified that this device does not affect the long-term viability of sensitive cells and are using this device to load proteins into macrophages to study the sensitive cells' signaling pathways.

The improvements we made to the test platform in FY 2004 (including the use of a four-electrode system, the development of electrical circuitry and software for on-chip integration, and the integration of a pressure transducer) were compatible with this new device. Together, these various improvements allow consistent delivery of electroporation signals to a single vesicle and acquisition of electrical and fluorescent evidence of electroporation.

The culmination of this effort demonstrates a practical and simple solution for handling a picoliter biomimetic volume on-chip by synthesizing robust vesicles and applying electrically monitored electroporation.

Significance

Despite the wide parameter space accessible in the design of chip-based microfluidic devices, important constraints remain, including the effects of diffusion and of inhomogeneous velocity profiles in pressure-driven flow, which can cause unwanted mixing or dilution of analytes. In this project we invented a sample management scheme to circumvent these issues by keeping analytes in containers on a size scale comparable to device features, which can be opened when mixing and diffusion are desired and closed during transport and analysis.

We employ lipid bilayer "giant" vesicles as picoliter reaction containers because they are in the size range (10-20 micron diameter) equal to common microfluidic channel dimensions and so can be manipulated and analyzed individually. We showed that these giant vesicles can be fabricated in a manner compatible with integration into a microfluidic device and can withstand expected types of manipulation in such a device.

We designed and mechanically tested new formulations of phospholipid vesicles that can withstand the conditions necessary to make them useful as containers in microfluidic devices. We showed that vesicles can be loaded with gel precursors or fluorescent dye, and that material from a specific vesicle can be controllably exchanged with the environment when a trapped vesicle is electroporated. The vesicle can be moved by pressure-driven flow, trapped on a microfluidic electroporation chip, and electroporated multiple times without failure, allowing current to flow through it and analytes to be exchanged in the process.

The techniques developed in this project can be applied to study living cells at the single cell level. Currently, our test platforms are being used on Sandia projects to study individual cells and ion transport in nanochannels. The culmination of this effort provides the appropriate biomimetic materials and technology needed to intelligently manage samples for more sensitive and accurate nanodetection.

There are many applications for such a system: in biochemistry as a platform for the study of molecular dynamics, in chemical/biological threat detection as a dilution-free conveyance and preparation motif, and in medicine as an accurate and local drug delivery technique. The advancements attained in this effort constitute an important step forward in the development of technology for manipulation of picoliter volumes of material.

Refereed Communications

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Diatoms as Molecular Architects

79821
B. A. Simmons, S. Aubry, D. Robinson, P. Lane, T. W. Lane

Project Purpose

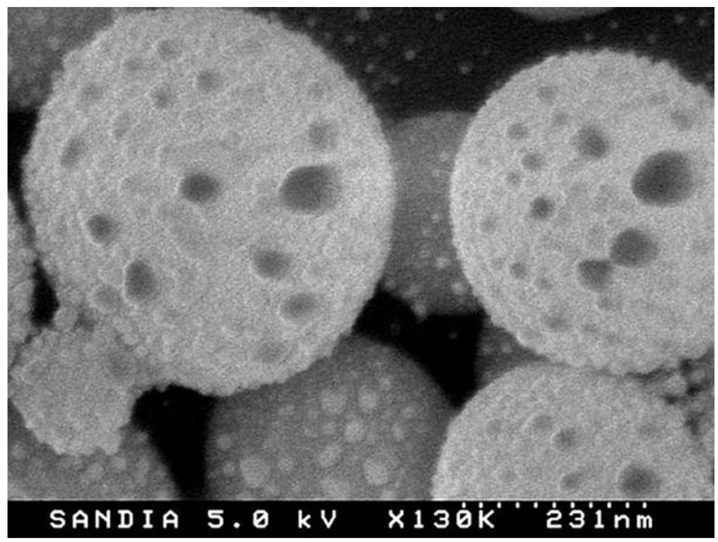
Diatoms are eukaryotic algae that are ubiquitous in marine and freshwater environments. Their single most compelling characteristic is that each cell is surrounded with a pillbox-like outer shell called a frustule that is formed of highly structured amorphous polymerized silica. Diatoms are able to transform soluble silica into these sturdy intricate structures at ambient temperatures and pressures. The degree of control and exquisite reproducibility by which the diatoms carry out this activity greatly exceeds current state-of-the-art manufacturing and any known biochemical capabilities in both industry and academia.

Understanding and ultimately controlling these specific biological and chemical processes would allow scientists to generate complex hierarchical structures on demand with a wide range of technological and industrial applications. The complex structured silica shell, or frustule, is the fingerprint used to differentiate the more than 10,000 species of diatoms known to the scientific community. At the basic level, there are two types of frustule macromorphologies: pennate, displaying bilateral symmetry; and centric, exhibiting radial symmetry. There are many permutations on these two basic forms and the micromorphology of the frustule can be quite complex, with all types of pore arrangements and morphologies.

It is our goal to add to the knowledge already generated in the scientific literature and attempt to identify the key factors that control the silica deposition process, and then use this knowledge to generate complex silica structures in a highly controlled and predictive fashion in a laboratory setting.

FY 2006 Accomplishments

We have met and exceeded our FY 2006 milestones. We extended the amine catalyzed silica polymerization and materials science effort into the realm of confined environments and determined their impact on the silicate structures. Through the use of reverse micelles as reaction containers, vesicle bilayers as artificial cellular membranes, and zinc oxide multilevel structures as peptide directing agents, we quickly realized significant accomplishments across the entire spectrum of advanced materials.



Electron micrograph of silica particle after calcination showing inner porosity of aggregate structure

The computational effort completed the initial steps toward a fundamental investigation of the molecular length scales involved in amine catalyzed silica polymerization. This involved the creation of a robust numerical system with unique inputs that can encompass the relevant chemical species terms and relevant time scales. The biotechnology effort is poised to impact the proteome and genome of several different diatom species to determine the exact sequence of events involved in the natural system. This will pave the way for the eventual genetic modifications of the diatoms to create tailored multilevel silica structures in a tailored fashion.

We received beam time at the low-q scattering instrument at the Los Alamos Neutron Scattering

Center and the grazing-incidence small-angle x-ray instrument at Argonne National Laboratory to elucidate the impact of interfacial chemistry on the silica polymerization process. And we submitted two technical advances in the field of silica polymerization.

Significance

The pursuit and realization of bioinspired advanced silicate materials is essential to the success of the DOE Office of Science objectives for molecularly tailored nanostructured materials. The science and technology produced by this project will benefit that objective by merging the realms of bio- and nanotechnology to tailor the morphology and functionality of those materials. This research is expected to impact the fields of next-generation sensors, catalysts, and composite materials.

Refereed Communications

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Other Communications

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Novel Mechanisms of Nanomechanical and Transmembrane Actuation

79823

K. Leung, S. L. Rempe, M. E. Chandross, K. R. Zavadil, J. A. Shelnutt, C. J. Medforth

Project Purpose

In nature, as in synthetic "smart" materials, local energetic interactions propagate over distances to do mechanical work. Harnessing such energy transduction mechanisms will impact future national security concerns ranging from synthesizing robust, integrated nanosized devices and manipulating nanoparticles on surfaces to preparing new types of sensors.

Synthetic nanotools based on nickel porphyrin triggers are among the simplest energy transduction prototypes. Photoexcitation or chemical binding changes the effective size of the nickel ion, inducing motion in the tightly bound porphyrin ligand and causing nanomechanical motions that can be amplified via specifically tailored structures attached to the porphyrin, creating nanotweezers. Tightly bound metal-ligand interactions are also believed to participate in the actuation (i.e., opening/closing motion) of biological transmembrane ion channels. These channels transmit or block electrical signals in response to gating events that can be strongly correlated with channel conformation and selective ion binding.

X-ray data exist for the closed KcsA configuration, but are "snapshots" and do not fully reveal the complex actuation mechanism. Since certain underlying principles of transmembrane proteins and the simpler porphyrin systems appear similar, and because nickel-porphyrin actuators can potentially be integrated into ion channels to perform gating in response to environmental changes, we are combining computer modeling assisted synthesis, x-ray crystallography, nuclear magnetic resonance, Raman, ultraviolet-visible absorption, and quantum spectral simulations to obtain a clear understanding of nanomechanical motion in robust, photo- or chemotriggered porphyrins.

We are developing theoretical techniques to treat actuation-induced nanomechanical motion using porphyrins as test cases and extending them to study metal ion (e.g., Na, K, Tl, Rb) size-induced conformational changes in ion channels. The scientific understanding revealed by studying these two complementary systems will impact smart materials and molecular electronics synthesis, nanoparticle manipulation, and national security needs for biosensors.

FY 2006 Accomplishments

We adapted a porphyrin-specific force-field to model the actuation of the porphyrin nanotweezers (NiBCP-8) as they switch between the low-spin nickel open $\alpha\beta\alpha\beta$ and the high-spin closed $\alpha\alpha\alpha\alpha$ structures. We discovered that rotation of one chain-bearing cyclopropyl substituent on $\alpha\beta\alpha\beta$ forms a stable intermediate $\alpha\alpha\alpha\beta$ structure. Then, rotation of the substituent carrying the other chain closes the nanotweezers and produces structure $\alpha\alpha\alpha\alpha$. This establishes the mechanism of nanotweezer actuation.

Experimentally, our collaborators at the French Atomic Energy Commission investigated the species formed when nanotweezers were dispersed in piperidine using circular dichroism. We detected a mixture of unligated low-spin open $\alpha\beta\alpha\beta$ form and the ligated high-spin closed $\alpha\alpha\alpha\alpha$ forms. This provides the first direct experimental evidence for tweezer closing in solution. The activation energy for tweezer opening and closing was estimated to be < 24 kcal/mol.

We successfully deposited several metalloporphyrins from benzene directly onto Au(111) substrates. Low-current scanning tunneling microscopy (STM) clearly indicates that cobalt tetraphenylporphyrin (CoTPP) molecules do not significantly perturb the surface. The presence of CoTPP can be confirmed by voltametry potential cycling, which produces no discernable nanoscale morphological changes, indicating that the film is stable under the conditions used.

We investigated the potentially porphyrin-coordinating 11-amino-undecanethiol self-assembled monolayer as an alternate substrate for nanotweezers. Low-current STM imaging reveals smooth continuous films with a low density of defect regions. We estimate that 25 to 33 percent of the alkyl pendant projection groups are aligned normal to the surface, yielding a good topographic platform for attaching nanotweezers. Atomic force microscopy has successfully been used to detect 1 micron wide stripes of the alkylthiol deposited using microcontact printing techniques.

We conducted DFT+U-based ab initio molecular dynamics simulations and found that the Mn(III)P-NO complex rapidly dissociates in water, while Mn(II)P-NO is long-lived. Both Mn(II)P and Mn(III)P strongly bind to NO^-. This confirms that electrochemical switching of MnP in water can distinguish NO and NO^- and suggests another sensor application for deposited nanotweezers.

We successfully created a model of the potassium ion channel selectivity filter, using a density functional theory treatment of the channel structure determined by x-ray crystallography and a dielectric treatment of the more distant protein or water. We discovered that local channel flexibility in the selectivity filter controls ion selectivity. In the presence of a permeant ion (e.g., K^+), the selectivity filter gate is open and the channel is in the ion-conducting state. With a nonpermeant ion (e.g., Na^+), the channel denatures and closes the selectivity filter gate, leaving the channel in the nonconducting state.

We further discovered that, in all models of explicit molecular ligands (water, formaldehyde, diglycine), ion coordination preferences characteristically shift to high values (6 for Na^+ and 8 for K^+) in very low dielectric environments (~ 1). Furthermore, the 8-coordinated ligand structures around K^+ for all molecular models (water, formamide, diglycine) optimize to nearly the same skewed cubic geometries revealed in x-ray crystallography of the KcsA selectivity filter binding.

Significance

Nanotweezer Deposition on Electrodes and Actuation Motion

The solution-phase nickel nanotweezer research lays the groundwork for deposition and integration of these smart molecules on to material support. By modifying the porphyrin substituents and choosing an appropriate ligand, solvent or surface, it should therefore be possible to fine tune the stabilities of the different structures to produce versatile nanotweezers. Our experimental work will facilitate such technological breakthroughs.

Deposition of Nanotweezers on Gold Electrodes

Our work in this area is leveraged by several other projects, including one that demonstrates the binding of ferrocene molecules on cyclohexadextrin-coated electrodes. It seeks to integrate biomolecular recognition and actuation mechanisms onto material support, especially on metal electrodes where electric potential cycling can be used to detect or release molecules. The resulting "smart" composite material surfaces can potentially be used as biosensors, and might be triggered to change the hydrophobic/hydrophilic characteristic of a nanofluidic channel governing water/protein passage. This will, potentially, strongly impact Sandia's mission and homeland security needs and may lead to future funding in the area of nanoscale optoelectronics applications.

Modeling Manganese Porphine-Nitric Oxide Complex Dispersed in Water

Nitric oxide has important physiological functions; an entire journal is devoted to its biochemistry. Manganese porphyrins dispersed in water or coated on electrodes have recently been demonstrated to detect NO in electrochemical settings and to distinguish it from NO^- and HNO. Our modeling of the dissociation of the manganese(II) porphine-nitric oxide complex in water marks the first time ab initio molecular dynamics simulations of porphyrins in the aqueous phase have been successfully carried out.

Furthermore, this work exploits the DFT+U approach we pioneered and published during the first year of this project, which will likely strongly impact the computational physics community in the modeling of transition metal complexes critical to biology and catalysis applications. This should have a significant impact on the scientific community at large.

Potassium Ion Channel Modeling
Our work on ion coordination structure and hydration free energies in aqueous and protein environments leveraged a desalination project that seeks to remove sodium and chloride ions in brackish water to create a cheap source of drinking water and an LDRD Project (90493 "Exploiting Interfacial Water Properties for Desalination and Purification Applications") that considers the fundamental issues of water and electrolyte interactions at material interfaces.

Refereed Communications

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Carbon Nanotube Sorting via DNA-Directed Self-Assembly

79824

A. L. Frischknecht, K. Leung, S. L. Rempe, P. M. Dentinger, M. G. Martin, D. Robinson

Project Purpose

Single-wall carbon nanotubes (SWNTs) show great promise for novel applications in molecular electronics, biohazard detection, and composite materials. Carbon nanotubes exhibit phenomenal mechanical strength and environment-sensitive electrical properties that range from metallic to semiconducting, depending on tube diameter and helicity. Commercially synthesized nanotubes exhibit a wide dispersion of geometries and conductivities, and tend to aggregate. Hence, the key to using these materials is the ability to solubilize and sort carbon nanotubes according to their geometric/electronic properties.

Recently published experimental work demonstrated that single-stranded deoxyribonucleic acid (DNA) will bind to carbon nanotubes, solubilize them in water, and allow sorting of the nanotubes based on their diameter. However, the mechanism for this is not currently understood and most interestingly, depends on the DNA sequence.

The purpose of this project is to understand DNA/carbon nanotube binding and its dependence on DNA sequence and the solution environment. This is a very difficult problem to attack, and so to date we have focused on two areas: 1) understanding the interactions between individual nucleic acid bases and nucleotides with carbon nanotubes using experimental and theoretical methods, and 2) developing algorithms that are more capable of simulating complex bio-molecules such as DNA.

One goal of this work is to discover whether the binding of nucleic acid bases depends on the electronic structure of the nanotubes, i.e., on whether the tube is metallic or semiconducting. Another is to find out whether the binding energy differs among the different nucleotides and how that energy depends on the salt concentration in solution.

Somewhat more long-term goals are to simulate a longer DNA molecule binding to a nanotube in order to explore the configurations and energetics of such binding. Ultimately this work will impact Sandia's mission in science-based materials processing, novel sensors for homeland security, and control of nanosystems.

FY 2006 Accomplishments

A major accomplishment was the completion of a new configurational-bias Monte Carlo algorithm, which is, for the first time, capable of efficiently sampling complex molecules, such as nucleotides, and of reliably regrowing cyclic molecules, such as the nucleic acid bases.

We also continued our study of the interactions between individual nucleic acid bases and nucleotides with SWNTs. Classical simulations of the binding of six different nucleotides to SWNTs have been performed, in water and with counterions and added salt. We found that the dynamics of ion association are very slow, in agreement with recent simulations of DNA. The slow dynamics have necessitated very long MD simulations, up to at least 50 ns. We determined the enthalpy of binding and found a marked salt effect for some of the nucleotides, which appears to be related to the conformations of the nucleotides near the nanotubes.

At smaller length scales, we performed ab initio calculations of the binding energy for water both inside and outside a SWNT. Calculations of the binding energy for nucleic acid bases are also proceeding, and we are comparing these to the classical results. Electronic density functional theory calculations show that the nucleic acid bases bind only weakly to SWNTs and do not undergo any charge transfer reactions with the SWNT. Any effects of DNA on the electronic properties of the SWNTs must therefore take place on larger length scales and do not appear to be quantum effects.

This work was complemented by an experimental effort in which we studied the adsorption of nucleotide monomers to nanotubes in aqueous salt solutions. We studied the degree to which nucleotides adsorb on nanotubes as a function of their relative weight fractions, temperature, equilibration time, and nucleotide type. We fit our data to a Langmuir isotherm, yielding an estimated binding strength and saturation level.

We found that nucleotides with larger rings and more electron-donating functional groups adhere more strongly to the tubes, and that nucleotides that adhere more strongly are also more effective at breaking up aggregates of tubes, thus creating additional surface area, allowing more material to adsorb. This effect was more noticeable at longer equilibration times.

Significance

The significance of the new Monte Carlo algorithm is considerable. This algorithm improves sampling for a large variety of molecules and thus will be generally useful to the molecular modeling community. This new algorithm is implemented in the Sandia-developed open source code Towhee, and is already being used by other researchers, and will be useful to a broad range of future simulation projects.

The classical simulation results are longer than many in the literature and represent one of a small number of studies examining ion dynamics near nucleotides in aqueous solution. These results are of value both to us and to the wider community in terms of giving a benchmark estimate for how long such simulations must be run to obtain reliable results. Thus in general, this project has enhanced Sandia's molecular modeling capabilities.

The experiments have added to our understanding of the surfactant properties of nucleotide monomers and of their interaction with carbon nanotubes. Such an understanding is likely to contribute to the ability to process carbon nanotubes in solution, facilitating chiral separation of tubes and assembly of tubes into useful, ordered structures.

Refereed Communications

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Next-Generation Contact Materials for High-Reliability Microsystems Devices

79825

P. T. Vianco, R. S. Goeke, J. A. Knapp, M. T. Dugger

Project Purpose

Dynamic electrical contacts are essential to microelectromechanical systems (MEMS) such as environmental sensing devices (ESDs), switches, and radio frequency (RF) devices. However, microsystem electrical contacts present unique materials challenges because, at the low forces associated with MEMS devices, actual contact occurs at a few surface asperities. Microwelding can stick the contacts together. Also, excessive oxidation and the decomposition of contaminant films can potentially increase contact resistance.

The goal of this project is to understand the effects of contact force, electrical current, and operating environment on contact resistance and adhesion properties of MEMS electrical contacts and then to correlate the resistance and adhesion behaviors with fundamental degradation mechanisms such as microwelding, surface oxidation (kinetics), contamination build-up, and changes of contact material properties.

We are using two approaches to understand the mechanisms that degrade contact performance. First, mixed metal coatings are being created using solid-state diffusion processes between multiple thin film stacks. Annealing treatments are providing coatings of different compositions and thus different adhesion and contact resistance properties for identifying degradation mechanism(s). The second approach explores the contact resistance and adhesion properties of passivation layers and novel transfer coatings.

Although the latter layers are more likely to resist microwelding, contact resistance may be degraded by surface asperities and organic contaminants. These potential degradation mechanisms are not currently understood. As part of these investigations, we are developing a test methodology that includes procedures for measuring fundamental coating properties as well as a MEMS test structure for characterizing long-term reliability. Lastly, we are exploring computational techniques with which to model surface interactions.

The objective of this task is to understand the capabilities of numerical models to predict contact resistance and sticking properties versus surface asperities and material properties (yield strength, melting temperature, and so on).

FY 2006 Accomplishments

We investigated a second series of multilayer thin films for creating robust electrical contact layers. The distribution of the Ge dopant species in the gold was difficult to control. The resulting film showed significant hardening, but the contact resistance was poor due to the surface oxide. We suspended the approach.

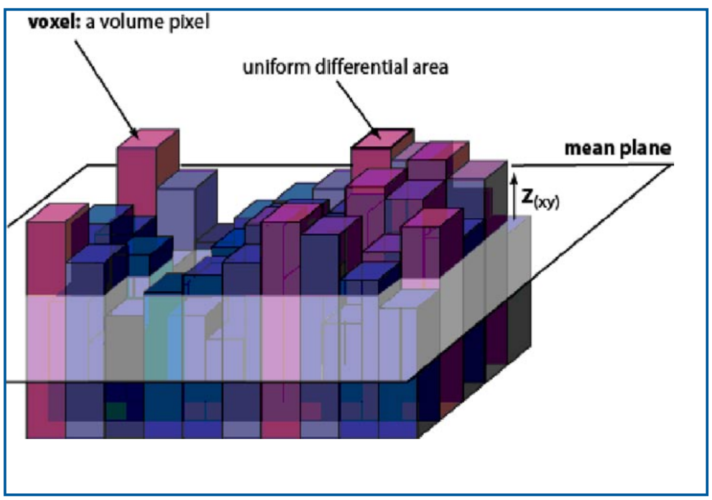
We examined several passivation layers for gold contacts. They included alkylthiol monolayers attached via sulfur linkage to the gold surface, tungsten disulfide films deposited by atomic layer deposition (ALD), and titanium nitride films grown by sputtering. Ultrathin (2-4 nm) TiN layers on Au reduced adhesion by 80 percent compared to the Au-Au contact alone. However, the TiN layer contact resistance was several orders of magnitude greater than Au-Au contact. Films of tungsten-disulfide 100 nm thick eliminated adhesion between the Au contacts. Resistance, however, was unacceptably high.

Alkylthiol self-assembled monolayers reduced adhesion significantly compared to untreated gold. They also yielded electrical contact resistance that was slightly better than untreated gold. The disadvantage to these types of films is that they have limited thermal stability and could degrade in potential packaging thermal exposures or during long term exposure to air.

We identified strong candidate contact materials in nanocomposites of gold with hard particle phases. We investigated low adherence and high electrical conductivity for codeposited gold and the secondary phases Al_2O_3 and TiN. These materials were created using pulsed-laser deposition (PLD) and sputter deposition methods. We developed a resistance network model to predict the resistivity of matrix/particle films in order to optimize the film composition for low contact resistance and minimum adhesion.

We designed a simple cantilever electrical contact test device to serve as a microsystem experimental platform for evaluating the performance of these composite films.

We overcame significant hurdles in developing a computational model of the electrical contact behavior of PLD Au alloy films. We obtained materials properties and topological measurements as model input data. We developed a three-dimensional surface model, based upon the height and contact of surface voxels. The model was exercised for an experimental surface and a prediction was made of the electrical contact area for that surface.



A voxel surface constructed from a profilometer-obtained data scan.

Significance

Our findings provided a new pathway for engineering thin films for the electrical contacts of MEMS devices. The PLD films of a gold matrix and alumina particles can minimize both electrical contact resistance and adhesion forces. The simultaneous realization of both of these attributes has been a challenge for current MEMS switch and sensor technologies.

In addition, the nonreactivity between these two components assures the long-term stability of the contacts that would be required for MEMS devices to be used in the nuclear weapons stockpile. The concurrent development of a modeling toolset will provide the means to predict contact behavior in order to minimize the need for extensive empirical studies, thereby minimizing the cost and time to optimize this thin film technology.

Refereed Communications

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Other Communications

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Controlled Fabrication of Nanowire Sensors

79826
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Project Purpose

Through an integrated multidisciplinary approach, from nanowire synthesis and device assembly to theoretical modeling, we are working to develop a new class of sensors with nanowires as the active elements. This new class of sensors is based on dramatic changes to nanowire conductivity when analytes interact with their surface because of the high nanowire surface-to-volume ratio. The purpose of this project is to develop the basic science understanding behind these nanowire sensors.

We are developing the understanding and methodologies to grow nanowires of different compositions and functionalize them with various organic functional groups in order to tailor their response to specific agents. On the fabrication front, we are exploring various approaches to assembling nanowire devices and performing sensing measurements. Theoretical modeling is used to understand and guide the development of these nanosensors.

FY 2006 Accomplishments

We synthesized novel metallic and semiconducting nanowires (Ge, Zn, ZnO), including some with very high aspect ratios. We assembled single-nanowire devices with carbon nanotubes, ZnO, GaN, and core/shell GaN/InN nanowires. A major improvement is the development of a method to fabricate top contacts, an important step for preventing analytes from affecting the nanowire/metal interface.

In addition, we used our nanoimprint lithography capability to fabricate arrays of Si nanowires and form transistors with them; we developed a method to form close-packed arrays of metallic nanoparticles between electrodes, which allows the replacement of ligands in-place; and we are developing a method based on enzymatic amplification for deoxyribonucleic acid (DNA) detection.

One important accomplishment is the demonstration of sensing in several of our devices. For example, we showed that the Si nanowire array is sensitive to solutions of nitrobenzene and phenol, with a response that is linearly dependent on the concentration. Another example is the demonstration that our carbon nanotube devices are sensitive to hydrogen gas, even at very low hydrogen pressures. In the area of optical sensing, we partnered with the University of Wisconsin to functionalize carbon nanotubes with chromophores, and we showed strong current modulation upon exposure to ultraviolet light.

On the modeling front, we developed a nonequilibrium quantum transport approach to calculate the current-voltage characteristics of carbon nanotube devices under illumination. We also obtained a detailed understanding of the effects of analytes on the conductivity of the Si nanowire array. This model allowed us to explain the linear dependence of the threshold shift in the output characteristics on the analyte concentration in the solution and to extract the binding energy of the analytes to the surface of the nanowires.

Significance

We developed a new method based on nanoimprint lithography to fabricate arrays of Si nanowires and showed that this array can be used for sensing gas and liquid phase analytes. To our knowledge this is the first such Si nanowire array fabricated using a top-down approach. Theoretical modeling explains the sensitivity improvement over planar devices and gives an approach to extract analyte binding energies from experimental measurements, which should be useful for the sensor community in general.

We also demonstrated sensing of hydrogen with carbon nanotubes and identified the mechanism for the sensing action, which has been an open question in the scientific community. Theoretical modeling provided a fundamental understanding of contacts between nanowires and metals, which will provide guidance for researchers in the scientific and nanotechnology communities.

We also developed new routes to synthesize ZnO and Ge nanowires and integrated them into our measurement platform for electrical characterization. In conjunction with the other nanowire materials that we have, this will provide for a suite of materials that can be functionalized for sensing. In collaboration with University of Wisconsin, we demonstrated that carbon nanotubes can be functionalized with chromophores and used as nanoscale optical sensors. Since different chromophores respond to different optical wavelengths, this opens the possibility of fabricating wavelength-specific optical detectors.

We also developed a new approach to performing measurements of electronic transport through molecular systems and used the approach to demonstrate sensing by replacing the linker molecules in place. This approach is compatible with spectroscopic diagnostic tools, allowing the simultaneous electrical and spectroscopic characterization of molecular systems. Such an approach is promising for surface-enhanced Raman scattering detectors.

Refereed Communications

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B. Simmons, A.A. Talin, E. Majzoub, and G. Lucadamo, "Surface-Engineered Metallic Nanoparticles and Their Utilization in Surface-Enhanced Raman Scattering (SERS)," presented at ACS Meeting, Washington, DC, August 2005.

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Fundamental Enabling Issues in Nanotechnology: Stress at the Atomic Level

79827

E. B. Webb III, S. M. Foiles, J. J. Hoyt, A. M. Morales, S. J. Hearne, J. A. Zimmerman, S. C. Seel

Project Purpose

To effectively integrate nanotechnology into functional devices, fundamental aspects of material behavior at the nanometer scale must be understood. Stresses generated during thin film growth can negatively influence component lifetime and performance. Stress has also been proposed as a mechanism for stabilizing supported nanoscale structures as well as tuning the optical response in III-V compound semiconductor materials. Despite the significant potential for engineering residual stress, the intrinsic connections between the evolving morphology of supported nanostructures and stress generation are still a matter of debate.

We seek to discover these fundamental connections so as to better understand how they can be manipulated to achieve a desired state of residual stress. We apply fully atomistic simulations to predict stress generation mechanisms and magnitudes during all growth stages, from island nucleation to coalescence and film thickening. We validate simulations by electrodeposition growth experiments on patterned substrates, which can establish the dependence of microstructure and growth stresses on process conditions and deposition geometry.

Sandia is one of the few facilities with the computational resources to perform atomistic modeling of stress generation mechanisms during thin film growth. This makes us well poised to discover fundamental mechanisms and understand how they may be engineered.

Atomistic simulations are exploring surface stress induced pressure (Laplace pressure) for isolated nanoislands in order to better understand the role of film/substrate binding in determining the stress in supported islands. We study island coalescence stress for island radii up to 0.25 micron with atomic scale detail; results are revealing structural changes associated with growth stress as a function of deposition conditions.

Deposition simulations onto surfaces intersected by grain boundary structures are permitting investigation of stress evolution during later growth stages, e.g., continual island coalescence and adatom incorporation into grain boundaries. The predictive capabilities of simulation permit direct determination of fundamental processes active in stress generation at the nanometer scale while connecting those processes, via new theory, to continuum models for much larger island and film structures.

Our results are revealing the necessary materials science to tailor stress, and therefore performance, in nanostructures and, eventually, integrated nanocomponents.

FY 2006 Accomplishments

Electrodeposition experiments for Ni on patterned and unpatterned substrates permitted the first direct investigation of stress generated from the onset of island coalescence as compared to stress generated during ongoing coalescence. This distinction is important in shaping theories for predicting stress/morphology relations and our experimental results confirm a significant degree of tensile stress is generated due to ongoing rather than initial coalescence, in contradiction to a number of existing theories.

We developed a new analytical model for describing island coalescence that permits a mechanistic interpretation of the phenomenon and produces predictions of stress magnitude in better agreement with experiment than prior models. We created new capability in Sandia's atomistic simulation code to model islands on surfaces with user-defined epitaxy relation and island/substrate interaction strength. This permits more direct comparison to experiment for a broader range of material combinations.

Using the new capability, we performed simulations to calculate stress in isolated islands and during island coalescence for varying substrate interaction strength. We prepared samples to attempt the first experiments to image free thin films (membranes) where the film thickness is about 100 nm and the average grain size is 100 to 1000 nm.

If successful, imaging the backside of the membrane during deposition on the front of the membrane will permit a direct, atomic scale, experimental investigation of existing theories for compressive stress generation during later stages of film growth. Experiments are directly coupled to simulations, which have already revealed unique adatom behavior near grain boundaries. This coupling permits a one-to-one correspondence on atomic mechanisms of stress generation.

Significance

The potential for precisely engineering stress in final thin film components is great in that it provides a direct path for avoiding unwanted failure (due to bending, warping, delaminating) or achieving some desired response (optical, electronic). This potential has driven a great deal of prior research into stress generation mechanisms but experiments and continuum theory lack the resolution necessary to unequivocally establish atomic scale behavior correlating to observed stress data during growth.

Ours is one of only a few groups in the world combining experiment and atomic scale simulation to overcome this barrier to understanding. Our work examining coalescence stress provides experimentally validated descriptions of the contribution to stress from initial coalescence compared to ongoing coalescence. This directly addresses a standing debate in the thin film community and provides initial understanding of how one can control the magnitude of tensile stress generated by coalescence via morphology control.

Our work examining later stages of (compressive) stress generation is also directly aimed at resolving a debate as to the role of grain boundaries and deposited adatoms. We confirmed an existing theory proposing adatom incorporation into grain boundaries as being a primary compressive stress generation mechanism. In addition, we designed a unique experiment to further explore this theory as well as to validate our numerical simulations.

The significance of this work is highlighted by a fairly significant number of invited presentations at thin film growth conferences. Through this research, important knowledge is being discovered to advance us toward more deterministic control of residual stress in material devices and components.

Refereed Communications

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Other Communications

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E.B. Webb III, "Atomistic Simulations of Stress Generation during Thin Film Growth," presented (invited) at the Gordon Research Conference on Thin Film and Small-Scale Mechanical Behavior, Waterville, ME, August 2006.

H.E. Fang, E.B. Webb III, S.C. Seel, J.A. Zimmerman, and J.J. Hoyt, "Thin Film Stress Dependence on Film/Substrate Interfacial Strength," presented at the World Congress on Computational Mechanics 7, Los Angeles, CA, July 2006.

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E.B. Webb III, S.C. Seel, J.J. Hoyt, and J.A. Zimmerman, "A New Model of Stress Generation during Thin Film Growth," presented at the Annual TMS Meeting, San Antonio, TX, March 2006.

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Effective Dispersion of Nanoparticles by Polymers

84266

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Project Purpose

Polymeric materials filled with dispersed nanoparticles show great promise for lowering the thermal expansion coefficient in nuclear weapon (NW) components and for use as high-resolution photoresists for lithography of novel microsystems. Also, the dispersion of nanoparticles is essential for a variety of other technologies important to Sandia, including high-dielectric-constant composites, ceramic processing, and solid-state lighting.

However, materials processing is a critical issue: stable nanoparticle dispersions are difficult to achieve, as traditional colloidal techniques are not effective for nanoparticles. Steric stabilization by attaching macromolecules to the particles has been shown to work in specific cases, but the underlying science is not well understood. In order to determine the optimal way to disperse nanoparticles in epoxies and other fluids, we are investigating the steric stabilization of nanoparticles by macromolecules.

We synthesize nanoparticles and attach polymers by functionalization or by polymerizing directly off the particle surfaces. We are exploring the effectiveness of various surface coverages, molecular weights, and charges for dispersing nanoparticles. The experiments and characterization provide input for and are interpreted using results from self-consistent field and classical density functional theory. Theoretical work includes predicting the structure of the polymeric layers and the interaction energy between coated nanoparticles.

The ultimate goal of this work is to enable the use of dispersed nanoparticles in nanocomposite materials for applications to NW components and microsystems, as well as other applications such as solid-state lighting and ceramics.

FY 2006 Accomplishments

We performed a number of calculations using a new density functional theory (DFT) code to determine that some of the materials synthesized in this project are in the mushroom regime (chains are not strongly stretched). We are building upon the DFT code to allow greater flexibility in modeling experimental systems of interest.

We devised a new titania synthesis that produces good quality nanoparticles of the rutile phase of titania at room temperature. Rutile is the highest refractive index phase of titania and is the desired phase for optical applications. The nanoparticles are stabilized by surfactants as-synthesized and therefore stay very well dispersed with no evidence of agglomeration.

We produced gold nanoparticles with polystyrene grown from the surface. We are determining the radial distribution function, $g(r)$, in this composite to inform and compare with theory.

We also developed a chemical method for dispersing commercially available titania nanoparticles. The method binds an amine terminated silane coupling agent to the surface, then reacts this with an epoxide functionalized silicone. The result is several nanometers of surface functionality that allows the titania to be easily dispersed in a silicone matrix.

Significance

We made great strides in the synthesis of narrow polydispersity titania particles and in dispersing them into polymers. Fully dispersed titania nanoparticles will enhance the refractive index of the polymer matrix without scattering light. This should permit tuning the refractive index of a polymer material to any desired value (limited on the low end by the polymer refractive index and on the high end by loading limits of the nanoparticles). This will allow much more versatile optics designs, as they will no longer be limited to discrete refractive index values, but will be infinitely tunable within a range of values.

There is also the issue of light extraction from light-emitting diodes (LED), a key consideration for solid-state lighting. The majority of light produced by current LEDs is lost to internal reflection in the diode chip. The high refractive index of the LED chip (~ 3.4 for GaAs) coupled with the low refractive index of the encapsulant material (typically 1.4 to 1.8 for epoxies or silicones) reflects much of the light that approaches the interface off-perpendicular.

The amount of reflected light scales approximately with the square of the critical angle for total internal reflection. Based upon this relationship, one can perform a quick calculation of the possible improvements based upon increasing the refractive index of the encapsulant.

Changing the encapsulant refractive index from 1.4 to 2.0 provides a doubling of light extraction, while an increase to 2.5 provides a tripling (based upon a GaAs LED). The improvements are even more dramatic for an LED with a lower refractive index like GaN, where a greater than five-fold enhancement is possible for a refractive index of 2.5.

Composites with refractive indexes this high are quite achievable using rutile in a highly loaded nanocomposite. This requires careful control of the surface chemistry and dispersion to achieve the high loading necessary.

Nanocrystalline Aluminum Alloys for Structural Applications

93528

C. W. San Marchi, D. J. Bammann, A. A. Brown, N. Y. Yang

Project Purpose

Bulk nanocrystalline (NC) alloys are an exciting new class of materials that have only recently established their potential for real-world applications. Nanocrystalline 5083 aluminum, for example, has twice the strength of conventional 5083, resistance to fracture near that of conventional high-strength aluminum, and the ability to resist loss of hardness following annealing at 500 °C for two hours. The NC-Al7Mg alloy (aluminum 7 wt% magnesium) has higher strength and ductility than the NC-5083 alloy, and has excellent thermal stability.

The excellent thermal stability obtained in cyromilled nanocrystalline aluminum alloys (NC-Al) is unique to material processed by cryomilling due to the highly refined nature of the microstructure. Typically, aluminum alloys cannot be used at elevated temperature, which is of particular importance for nuclear safety in the event of fire and may increase the maximum temperature that aluminum alloys can be implemented as structural components. The higher strength of NC materials typically comes at the cost of lower ductility; however, it has been demonstrated that large grains in a NC matrix can substantially improve the ductility without loss of the high strength contributed by the NC matrix.

We are studying microstructures of this type, called multiscale microstructures (MSM), with the goal of producing high-strength aluminum alloys with high ductility. The fundamental microstructural processes of precipitation, recrystallization, and deformation of NC materials, however, are not well understood.

The goals of this work revolve around understanding the physical and mechanical metallurgy of this new class of structural materials. With a greater understanding of the microstructure-property relationships in cryomilled NC-Al, we can create philosophies for engineering nanoscale microstructures for optimal performance with regard to strength and ductility, as well as thermal stability.

The development of constitutive tools will facilitate understanding the sources and thermal evolution of strength and ductility of these novel microstructures and will further illuminate strategies for advancing innovative NC-structural materials. We are developing the experimental and computation tools necessary to consider NC-Al for structural applications at ambient and elevated temperatures. Such tools are expected to have broad scientific impact on the study of NC materials and to spur the commercial application of bulk nanocrystalline materials.

FY 2006 Accomplishments

We made progress toward understanding the metallurgy of nanocrystalline aluminum produced via cryomilling, focusing on the 5083 alloy, since this system has received significant interest for military application. Our scope during this first year also included an aluminum-magnesium binary alloy. This binary alloy (Al-7Mg) is cleaner than commercial alloys, such as the 5083 aluminum alloy, and also has slightly superior strength when processed as a nanocrystalline material.

Comparison of the microstructures and properties of these alloys provides clues to the phenomenology of deformation and fracture in cryomilled materials. Our investigations, however, have raised numerous fundamental questions about nanocrystalline alloys, questions that have not been satisfactorily addressed in the literature, such as the role of inclusions and high-energy grain structures in governing mechanical response and thermal stability; questions that we are addressing by combining microstructural evaluation, a comprehensive testing effort, and constitutive modeling.

During this first year, we also developed a relationship with the Army Research Laboratory that will lead to exchange of technical and scientific information complementing our activities and potentially lead to new interest in nanocrystalline metals. To this end, we are working to upgrade capabilities for high-strain-rate testing. We also generated data from high-strain-rate testing with collaborators at Purdue University. These data are currently being analyzed.

Significance

Our study of nanocrystalline aluminum (NC-Al) is raising and addressing many questions about these unusual materials and nanocrystalline materials in general. Nanoscale phenomena in metals are not well understood, and these investigations are providing the experimental basis for developing this understanding. For example, we have observed unusual grain boundaries (often called nonequilibrium grain boundaries in the literature), but the role these boundaries have on thermal stability, strength, and ductility are not understood.

We documented in detail the microstructure of NC-5083 (5083 refers to a common composition of aluminum alloys), which is providing guidance to the microstructural modeling and simulation effort. There is a complementary project starting this year on modeling and simulation of nanocrystalline materials that will benefit from our analysis and measurements. We will work closely with those investigators to find synergies between projects.

Modeling and simulation are of particular importance to the study of nanostructured microstructures because it is very difficult to comprehensively study physical materials at the nanoscale, especially ones as microstructurally complicated as these. Computer experiments will assist in fundamental understanding using idealized microstructures that cannot be easily produced in the laboratory.

One of the major challenges with the study of cryomilled NC-Al is quantifying the microstructure, which consists of a wide distribution of grain sizes, large volumes of submicron inclusions, diverse grain boundary structures, and so on. Single values of grain size, for example, do not adequately describe the role of the varied nature of the grain structures. Thus, we are developing relationships with other groups at Sandia that are developing new techniques for studying the structure of materials at the nanoscale by combining information from several atom-scale techniques such as three-dimensional atom probe and transmission electronic microscopy tomography.

If NC-Al specimens can be prepared for these methods, we may be able to seek additional collaborations that will provide significant new information to this project and the NC community at large as well as provide a platform for the further development of these new techniques.

One significant example of a potential contribution to the study of NC-Al from our work relates to the interpretation from the literature that atomic scale nitride phases at grain boundaries may provide a significant amount of strengthening and thermal stability. Our preliminary experiments hint that the feature identified in the literature may be an artifact of electron beam damage during investigation.

The experimental mechanical testing results have generated significant interest, as well. The military is interested in these high-strength aluminum alloys for armor applications. Other applications being developed at Sandia could benefit from aluminum alloys that are thermally stable to temperatures near 200 °C or higher, for which no conventional aluminum alloys are considered appropriate.

Refereed Communications

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Other Communications

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Nanoparticle Flow, Ordering, and Self-Assembly

93529
G. S. Grest, J. B. Lechman, J. M. Lane, M. Horsch, N. S. Bell, P. R. Schunk

Project Purpose

Nanoparticles are now more than ever being used to tailor materials function and performance in differentiating technologies because of their profound effect on thermophysical, mechanical, and optical properties. The most feasible way to disperse particles in a bulk material or control their packing at a substrate is through fluidization in a carrier that can be processed with well-known techniques (e.g., spin, drip, and spray coating, fiber drawing, casting followed by solidification through solvent evaporation/drying/curing/sintering).

Unfortunately, processing particles as concentrated, fluidized suspensions into useful products remains an art largely because the effect of particle shape and volume fraction on fluidic properties and suspension stability remains unexplored in a regime where particle-particle interaction mechanics is prevalent.

To achieve a stronger scientific understanding of these factors, we are bridging scales between atomistic and molecular-level forces active in dense nanoparticle suspensions (e.g., colloidal forces) with hydrodynamic forces using numerical simulation and experimental validation. Our approach hinges on research and development of interparticle, particle-solvent, and particle-wall force models that account for a variety of chemical/steric/electrostatic effects. These models, together with a flow solver, will be deployed in an existing discrete-element-model software framework. With this new capability, the two most fundamental challenges in manufacturing can be addressed by creating stable nanoparticle dispersions for processing and controlling particle adsorption and tailoring packing on substrates.

Our research provides further understanding of dispersion/suspension rheology, dispersion stability, phase behavior, and surface modification. This work has long-term implications in materials research and development across Sandia.

The focus of our research is to develop a stronger scientific understanding of factors that control processing and stability of concentrated, fluidized nanoparticle suspensions. This involves a multiprong approach, combining atomistic and continuum level modeling with experimental studies. During FY 2006 we concentrated on researching and selecting a Navier-Stokes (NS) solver for the background fluid, which is numerically accurate and efficient, to incorporate into our present discrete element particle code.

We carried out extensive molecular dynamics simulations of nanoparticles in an explicit solvent to define the range of applicability of this code. We identified a prototype particle/fluid system for experimental validation and carried out basic rheological and dispersion stability for this prototype system.

FY 2006 Accomplishments

Numerical

We implemented a coupled NS solver and discrete-element-method (DEM) code that is capable of simulating dense nanoparticle-laden flows with realistic representation of fluid-particle interaction and contact mechanics. To increase the computational efficiency, the NS solver was parallelized and coupled to an existing parallel discrete-element-method code that has been used to successfully model discrete particles in the absences of a background fluid.

To validate this code, we compared the ratio of the drag force on a fixed test particle due to the presence of a neighboring particle to that of an isolated particle with experimental results. We carried out a series of simulations of flow through packs of spheres to further determine and validate the method's performance for multiple, closely spaced particle systems.

To assess the limitations of the NS/DEM methodology, we carried out multimillion atom simulations explicitly including the solvent in order to gauge the minimum size of a nanoparticle and bulk concentration of particles at which the continuum fluid-solver approach is valid. We conducted simulations for binary hard sphere mixtures of particles to determine the effective attractive interaction between the larger nanoparticles due to the presence of the solvent.

We also advanced the LAMMPS code to include the prototype pair-wise potentials for colloidal dispersions. Specifically, van der Waals and osmotic forces were included to model a system of sterically stabilized, bidisperse metal nanoparticles. We performed melting simulations in both two and three dimensions for lattices of large and small diameter particles. Using a simpler particle mechanics code, we also studied self-organization of nanometer- to micron-sized particles from a bulk solution into an adsorbed monolayer. Special effort is being given to understanding how hydrodynamic forces affect microstructural formation at a surface.

These studies will help us gain a greater fundamental understanding of how such forces affect pattern formation in colloidal systems, and in particular, how hydrodynamic forces can be used to provide an alternative to thermal or Brownian forces as a means of sampling configuration space.

Experiments

We considered a number of potential model nanoparticles systems that could be studied both experimentally and computationally. Nanoparticles composed of monodisperse (< 5% size variation) silica spheres, functionalized with polydimethylsiloxane (PDMS) polymer chains covalently bonded to the surface were chosen because of their use in a number of industrial applications and scientific studies.

We chose low-molecular-weight PDMS silicone oil as the solvent. These particles can be loaded in the fluid phase at increasing volume fractions for rheological measurements of the elastic and inelastic modulus. Computationally, the interaction potentials for atomistic simulations of PDMS are known and have been tested as part of a separate project on polymer adhesion. The synthetic effort was successful in modifying monodisperse silica colloids with a diameter of 284 nm with monoepoxide terminated PDMS chains. We characterized rheological properties as a function of shear rate for samples of varying volume content. We also determined elastic and loss moduli in oscillatory stress and frequency tests.

Significance

This first year we focused on developing and validating a mesoscopic-scale, nanoparticle/fluid modeling and simulation tool as an enabling technology to advance the development and commercialization of specialty nanocomposite materials manufactured from suspension-based processing. New materials containing nanoparticles are important for the enhancement of processing, physical, and aging properties of materials for next-generation commercial and defense applications. The results of this research are being leveraged be experimental and modeling research being carried out at the Center for Integrated Nanotechnology.

Refereed Communications

C.H. Rycroft, M.Z. Bazant, G.S. Grest, and J.W. Landry, “Dynamics of Random Packings in Granular Flow,” *Physical Review E*, vol. 73, p. 051306, May 2006.

O. Baran, D. Erta, T.C. Halsey, G.S. Grest, and J.B. Lechman, “Velocity Correlations in Dense Gravity Driven Granular Chute Flow,” to be published in *Physical Review E*.

Other Communications

J.B. Lechman, “Numerical Simulation of Granular and Multiphase Flow,” presented at Exxon Mobil Research and Engineering Co., Annadale, NJ, April 2006.

J.B. Lechman, “Numerical Simulation of Granular and Multiphase Flow,” presented at the Center for Nonlinear Dynamics, University of Texas, Austin, TX, February 2006.

G.S Grest, “Packing and Flow in Granular Materials,” presented at the Physics Dept. Seminar, Emory University, Atlanta, GA, October 2006.

Pulsed Power Sciences

Embeddable Shock Physics Sensors

67122
T. A. Friedmann, C. S. Alexander, R. E. Setchell, M. D. Knudson, J. Mason, J. Davis, T. J. Vogler

Project Purpose

We are developing miniaturized embeddable sensors for use in Z experiments to measure the velocity, stress (both longitudinal and transverse), and temperature histories of propagating compression waves in materials. Currently, observations of shock and compression experiments at Z are limited to optical techniques. For opaque materials, this provides information only at material interfaces, and wave interactions at these interfaces must be taken into account.

To directly obtain measurements of dynamic compression behavior within a material sample, ways must be developed to imbed small, nonperturbing sensors into materials of interest and to electrically measure the material response. This approach provides challenges both for the sensor design and for recording of the generated signal in the extreme electromagnetic environment of Z. The sensors must be capable of nanosecond temporal resolution with inherently (very) large electrical noise rejection. It is expected that data generated from these sensors will allow for the determination of material equation of state data and strength that can be used to validate models of materials behavior under extreme dynamic pressure environments

FY 2006 Accomplishments

In the third year of this project, we focused on producing targets for gas gun experiments with significant progress being made that allowed for a test shot on a gas gun with a target containing embedded temperature (200 Å thin film type K thermocouple) and stress (thin film Manganin®) sensors. The areas of progress were:

Gauge Design and Fabrication

At the beginning of the year we redesigned the thin film Manganin gauge to incorporate thin film copper leads to better define the active gauge area. This complicated the gauge fabrication but allowed for lower resistance gauges that could be measured in a simpler two-wire configuration. In order to produce impedance-matched 50-ohm gauges, we also modified the deposition process to include an in situ resistance measurement. This technique was especially useful in fabricating the gauges on rough alumina substrates.

Developed Means for Recording Electrical Gauge Signals

We performed two successful gas gun experiments using commercial Manganin gauges to gain experience with recording Manganin gauge signals. We obtained fast amplifiers to measure the millivolt signals expected from the thermocouple gauges. These amplifiers were tested using a commercial thermocouple on a Z shot. The results showed that the noise in the Z environment was too great to gain a meaningful signal but did not rule out their use on gas gun experiments.

Target Design and Fabrication

We expended a significant effort to design appropriate target packages and impactors that would exercise the gauges under varying load conditions to assess their utility. The initial target design was made to test the gauge survivability and data acquisition procedure. The gauges were embedded in fused silica. The impactor was designed to produce a slow ramp followed by a shockwave. We designed four different targets and impactors for follow-on tests to perform if the initial experiments were successful. We designed these targets with varying materials (CaF_2, fused silica, and alumina) and began fabricating gauges and the targets.

Initial Gas Gun Experiments
The gas gun experiment was successful in recording the gauge response but revealed a problem with shearing of the lead connections that correlated with the arrival of the shock front. We designed a strategy to increase the gauge survivability by incorporating impedance matched epoxy resign to pot the gauge leads.

Significance

This work ties to DOE basic science and defense strategic goals. Better experimental understanding of materials under dynamic compression is necessary to improve our knowledge of materials behavior in extreme nonequilibrium environments and validate theoretical codes for input into materials models for predicting the performance and future reliability of weapons systems.

Characterizing the Emissivity of Materials under Dynamic Compression

79877

D. H. Dolan III, M. D. Roderick, A. W. Shay, J. W. Gluth, J. Davis, J. Podsednik

Project Purpose

Temperature measurements are crucial to equation of state (EOS) development but are difficult to perform reliably. In the case of optical/infrared pyrometry, a large uncertainty comes from the fact that sample emissivity (the deviation from a blackbody) is unknown. In this project, we are developing coatings that can be used as emissivity standards. Once such standards are characterized, high-pressure EOS and phase transition studies will greatly benefit from accurate temperature measurements.

An important consideration in using an emissivity standard is the way in which the material changes under extreme temperatures and pressures. Such changes can be investigated through a combination of static and dynamic measurements. Static high-temperature emissivity measurements are under way in collaboration with the National Institute of Standards and Technology (NIST).

Dynamic measurements are also being developed to study how shock compression changes material emissivity. The latter measurements combine single-event impact experiments with the intense, broadband radiation from the National Synchrotron Light Source at Brookhaven National Laboratory. By investigating changes of material reflectance under shock compression, we can infer how sample emissivity would vary in a pyrometry measurement under similar conditions.

Our research will focus on low-emissivity coatings developed at Sandia, laser heating materials (such as rhenium and tungsten), and standard metals (such as aluminum and copper). A key element of this project is the operation of a gas gun at Brookhaven National Laboratory, requiring extensive collaboration between the two national laboratories. Although the synchrotron is a user facility, this type of single-event measurement has never been attempted before. By demonstrating the feasibility of such experiments in the infrared, we will lay the groundwork for future work in dynamic x-ray diffraction research.

FY 2006 Accomplishments

Following previous work in the field, we investigated rhenium silicide as a high-emissivity coating material. The results indicate that the Sandia-developed chromium/chromium oxide film is a far superior emissivity standard. Static high-temperature measurements (performed at NIST) indicate that the emissivity of chromium/chromium oxide increases with temperature, but there are some ambiguities due to oxidation; system improvements should eliminate this problem. To allow detailed discussions of the chromium/chromium oxide coating outside Sandia, a technical advance was submitted.

We made substantial progress in the area of dynamic reflectance measurements. A 3-inch gas gun was constructed and tested at Sandia then shipped to Brookhaven National Laboratory. The gun was installed on the VUV (visible-ultraviolet) ring of the National Synchrotron Light Source and retested to ensure proper performance. A series of nine reflectance experiments were performed using synchrotron light; despite a few practical difficulties, reflectance data was obtained in all but one experiment.

We investigated reflectance changes in shocked aluminum, copper, chromium, and chromium oxide films in the near- and mid-infrared domain. Preliminary analysis suggests that most of these materials experience a minor decrease (~ 10 percent) in specular reflectance upon shock compression. This finding supports the common assumption that ambient measurements provide a lower bound for the emissivity of a shocked material. Moreover, there is evidence that the Sandia high-emissivity coating does not degrade under shock compression, and thus may serve as a useful emissivity standard.

Significance

The direct significance of this project is that we have demonstrated a technique for estimating the emissivity of a dynamically compressed material. With such measurements, it is possible to more rigorously constrain temperatures calculated in infrared pyrometry, an immense boost to equation of state and phase transition studies. A specific case where this is important is for low-emissivity materials, which may serve emissivity standards for a broad range of temperature studies.

Beyond the direct project results, there are a number of indirect benefits that have much broader implications. This project represents the first use of synchrotron radiation in single-event, time-resolved measurements. With some improvements in fast infrared detection, such measurements could be applied to a wide range of spectroscopic studies using dynamic compression to trigger phenomena of interest (e.g., phase transitions). Furthermore, the demonstration of safe gas gun operation in a synchrotron facility lays the ground work for future experiments using x-ray beam lines, which have enormous potential for probing materials under rapid compression.

Other Communications

D.H. Dolan, S. Becker, and R. Hacking, "Temperature Measurements of Dynamically Compressed Materials," presented at Society for Experimental Mechanics, St. Louis, MO, June 2006.

Beyond the Local Density Approximation: Improving Density Functional Theory for High Energy Density Physics Applications

79878

M. P. Desjarlais, R. P. Muller, A. E. Mattsson, A. F. Wright, N. A. Modine, M. P. Sears

Project Purpose

The motivation for this work is the well-known deficiency of local density functionals in reproducing experimental band gaps for insulators. Typically the band gap is underestimated by one to several eV. When combined with finite-temperature applications, this underestimation of the band gap leads to higher populations of electrons in conduction states (higher ionization degree) and greater calculated conductivities.

The primary purpose of this project is to develop a finite-temperature, exact-exchange (EXX) capability within Sandia's density functional code Socorro. Socorro is a plane wave density functional code with periodic boundary conditions. The numerical algorithms associated with EXX are exceedingly complex, and one significant component of this work is the optimization of those algorithms. Another numerical issue is the treatment of integrable divergences that appear in the evaluation of the exchange integrals.

A working EXX capability also requires, for efficient handling of larger atoms, a method for generating compatible pseudopotentials. Establishing this capability at Sandia is an important part of our project.

Although exchange is handled "exactly" in this approach, the correlation part of the complete exchange-correlation functional is not, and considerable research has been devoted outside of this project to answering the question of what such a functional would look like. One objective of this project is to explore several possibilities for correlation functionals compatible with EXX.

FY 2006 Accomplishments

Calculation of the optimized effective potential (OEP) has been combined with the EXX functional to achieve a working finite-temperature, EXX capability in Socorro. Results of test calculations for silicon agree well with published results from other EXX calculations. Considerable effort went into the optimization of algorithms. Our method has proven to be very efficient and has been applied to larger systems than have any existing EXX codes.

We have developed a framework for implementing and testing sample OEPl functionals. We have tested this framework using an OEP based on EXX as well as a correlated wave function. We have demonstrated that EXX functionals give accurate reproductions of band gaps and excitation energies for simple atomic systems, e.g., neon, helium, beryllium.

We also demonstrated that the band gap difference between EXX and GGA or LDA (GGA: generalized gradient approximation, LDA: local density approximation) diminishes with increasing temperature. This is consistent with other work that shows a similar result within a finite temperature GW scheme.

Our pseudopotential generation capability has gone beyond the Krieger-Li-Iafrate (KLI) approximation discussed in previous reports. Because of uncertainty in the accuracy of the KLI approximation, we desired a capability to develop pseudopotentials that were entirely consistent with our exchange functional. We obtained software for generating pseudopotentials consistent with EXX functionals based on the work of Engel et al. [1] and have successfully generated EXX pseudopotentials for aluminum and silicon.

We developed an EXX compatible correlation functional (C1). EXX+C1 clearly gives a different total energy than EXX+GGA correlation and EXX+LDA correlation. The energies for EXX+C1 were close to the EXX+GGA. All of the total energies obtained with correlation are quite different from the energies obtained with EXX only. We implemented a robust method of handling the integrable singularity in the exchange integrals. The architecture of the Socorro code has been modified in a way that makes future implementation of hybrid functionals straightforward.

Significance

We now have a functioning exact-exchange (EXX) capability in Sandia's Socorro code and have the capability to build compatible exact-exchange-based pseudopotentials. Unlike previous methods of implementing the EXX approximation, this new approach has a computational cost similar to traditional density functional theory (DFT) methods and is applicable at arbitrary temperatures. We demonstrated this capability with unprecedented EXX calculations for systems with up to 216 atoms and at temperatures up to 12,000 K.

In addition, calculations for the silicon self-interstitial showed that EXX is a promising approach to avoiding errors in DFT properties of defects that arise from the unphysical overlap of defect states with the valence or conduction bands. This has direct relevance to the Qualification Alternatives to the Sandia Pulse Reactor (QASPR) research program.

Our results demonstrate the usefulness of EXX functionals in correcting band gap problems that cause an over-estimate of conductivity and ionization in finite-temperature DFT molecular dynamics simulations of warm dense matter. Such a correction allows higher-fidelity prediction of optical properties for high energy density physics applications.

The capability we now have in Socorro also places us in a very good position to explore hybrid functionals in the future. Hybrid functionals – extremely popular within the chemistry community – is a likely area for future research at Sandia.

[1] E. Engel, A. Höck, R.N. Schmid, R.M Dreizler, and N. Chetty, "Role of the Core-Valence Interaction for Pseudopotential Calculations with Exact Exchange," *Physical Review B*, vol. 64, 125111 September 2001.

Refereed Communications

R.P. Muller and M.P. Desjarlais, "Optimized Effective Potential from a Correlated Wave (OEP-GVB)," *The Journal of Chemical Physics*, vol. 125, 054101, August 2006.

R.A. Lippert, N.A. Modine, and A.F. Wright, "The Optimized Effective Potential with Finite Temperature," *Journal of Physics: Condensed Matter*, vol. 18, pp. 4295-4304, April 2006.

Other Communications

N.A. Modine, R.A. Lippert, A.F. Wright, R.P. Muller, M.P. Sears, A.E. Mattsson, and M.P. Desjarlais, "Iterative Optimized Effective Potential and Exact Exchange Calculations at Finite Temperature," presented at American Physical Society March Meeting 2006, Baltimore, MD, 2006.

N.A. Modine, "Nano is Big: A First-Principles Electronic Structure Viewpoint; Synergy Between Experiment and Computation in Nanoscale Science," presented (invited lecture) at Harvard University, Cambridge, MA, May 2006.

Thermophysical Properties of Shocked Water for Modeling Pulsed Power Switches and Other HEDP Systems

79880
T. K. Mattsson, M. P. Desjarlais, L. K. Warne

Project Purpose

Few factors influence a high energy density physics (HEDP) simulation as directly as the thermophysical models of matter used. Without high-fidelity models for the equation of state (EOS) and electrical conductivity (σ), macroscopic simulations can be qualitative, at best. The purpose of this project is to better understand and model the properties of water under conditions encountered in pulsed power systems.

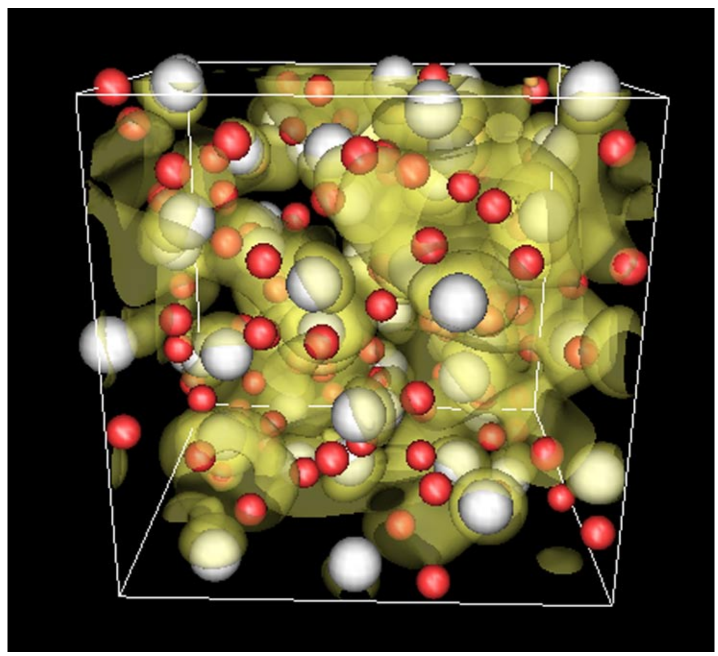
Water is being used as tampers in exploding wire experiments and in breakdown switches, for which understanding plasma channel conditions is a problem of great importance to future designs. In addition, experimental knowledge is limited due to the opacity of water; the breakdown channel itself is very difficult to study using optical methods, so a goal of the project was to verify materials models using density functional theory (DFT), state-of-the-art theoretical simulations for large and disordered systems. Specifically for FY 2006, the main objective was to develop an integrated model of conductivity (including ionic and electronic conduction) followed by finalizing the modeling over a wide range of temperature and density/pressure.

FY 2006 Accomplishments

In FY 2006, we continued the project mainly along the lines of the proposal: completing a quantum molecular dynamics database for EOS and conductivity for water, and using the database to develop wide-range EOS and conductivity models. Although the calculations of ionic conductivity proved more complex than initially expected, we succeeded in calculating electronic as well as ionic conductivity over a large range of temperature and pressure, a primary goal for the project.

A second main finding of the work is the strong demonstration that using a theory allowing for an electronic temperature is necessary when performing DFT in the field of HEDP. Although this has been the method used at Sandia for several years, it has not been uniformly agreed upon by researchers in the field that finite temperature DFT is required.

A third, most important, accomplishment is the discovery that the phase diagram of water should be revised in the high energy density region. These findings have been published and presented at international conferences and workshops and have been well received and publicized.



Structure of conducting water together with electron density from a partially occupied band.

Significance

The general significant of the project to the HEDP community is very high. Radiation-hydrodynamics simulations of high energy density physics are routinely performed in key DOE programs at Sandia, Los Alamos National Laboratory, and Livermore National Laboratory. The material models used for equation of state, electrical conductivity, and opacity limits the reliability of these simulations. Problems in the underlying models cannot be mended by improvements at higher levels: faster solvers, massive parallelization, or improved meshing. High-fidelity physics models, as developed in this project, are thus instrumental in reaching the goal of predictive modeling.

The specific findings for water are no less relevant; the new material model for water will be used in simulations at Sandia as well as shared with the national laboratories, DOE, and university partners. Simulations that will be made to a higher fidelity include shock waves in water, electrical break down in plasma channels in water, and the properties of giant planets, in particular Neptune.

Besides the direct impact of new, improved material models for water, a main conclusion of the work is the necessity to use a theory that allows for the electronic temperature when performing first-principles quantum simulations (using DFT) in the field of HEDP. Although this has been the method used at Sandia for several years, it has not been uniformly agreed upon by researchers in the field to use finite-temperature DFT. Our findings will therefore likely result in a permanent change in how HEDP simulations are performed in the future.

Refereed Communications

T.R. Mattsson and M.P. Desjarlais, “Phase Diagram and Electrical Conductivity of High Energy Density Water from Density Functional Theory,” *Physical Review Letters*, vol. 97, 017801, July 2006.

Other Communications

T.R. Mattsson and M.P. Desjarlais, “High Energy Density Water: Density Functional Theory Simulations of Phase-Diagram and Electrical Conductivity,” presented at 12th International Workshop on the Physics of Non-Ideal Plasmas, Darmstadt, Germany, September 2006.

T.R. Mattsson and M.P. Desjarlais, “First-Principles Simulations of the Electrical Conductivity of Shocked Water,” presented at International Workshop on Warm Dense Matter, Vancouver, BC, October 2005.

M.P. Desjarlais, “Quantum Molecular Dynamics Simulations in Support of High Energy Density Physics Experiments on Sandia’s Z Machine,” presented at GSI Colloquium at the 12th International Workshop on the Physics of Non-Ideal Plasmas, Darmstadt, Germany, September 2006.

M.P. Desjarlais, “QMD Simulations of Warm Dense Matter in Support of High Energy Density Physics Experiments on the Z Machine,” presented at Workshop on Accelerator-Driven Warm Dense Matter Physics, Pleasanton, CA, February 2006.

Triggered Low-Inductance Gas Switching

79881
H. C. Harjes, J. S. McGurn, J. P. Corley, J. M. Elizondo-Decanini

Project Purpose

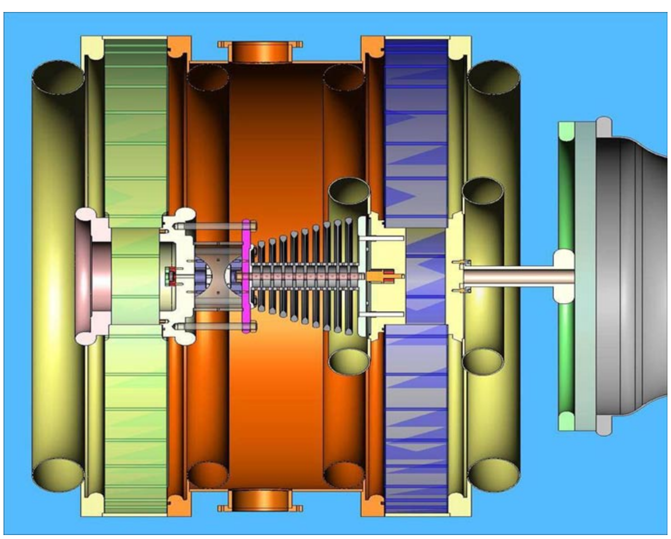
In present pulsed power systems, the laser triggered gas switch (LTS) is generally the primary switch. Methods for significantly decreasing the inductance of this switch would be of great value in improving system performance and possibly reducing system cost and complexity. In this project we are investigating methods of increasing the switch diameter while maintaining multichannel behavior and thus decreasing inductance.

In the first year of the project, we developed a switch design concept and started the design of a moderate scale 1.5 MV switch. In the second year, we completed the switch design and focused our effort on small scale multichannel breakdown experiments. We designed and constructed a test bed for testing 1 or 2 gap switches and began preliminary testing. In this test bed, multichannel behavior will be characterized as a function of voltage pulse rise time and electrode diameter. With this information, we can develop the design of a full-size 6 MV switch using larger electrode diameters to reduce inductance with greater confidence.

FY 2006 Accomplishments

When this fiscal year began, the design of a 1.5 MV switch was being completed. The cascade section of this switch had 10 gaps made up of electrodes of incrementally increasing diameters starting at the diameter typically used in proven switches and ending with a diameter twice that. After evaluating the hardware costs for the switch and the benefits of an experiment of this large scale, the project was redirected toward smaller scale tests (1or 2 gaps) to study and characterize the behavior of multichannel switch gaps.

We designed and constructed the small scale test stand, and preliminary test are under way. These early tests were performed with two 8-inch-diameter cascade electrodes (1 gap) from an existing switch in air at 1 atmosphere. These tests demonstrate the functionality of the test bed and diagnostics. Gap current and voltage measurements are recorded along with open shutter photos of the gap breakdown. Both single channel and multichannel breakdowns were been observed. Accomplishments for FY 2006 are summarized as follows:



Drawing of the assembled 1.5 MV switch.

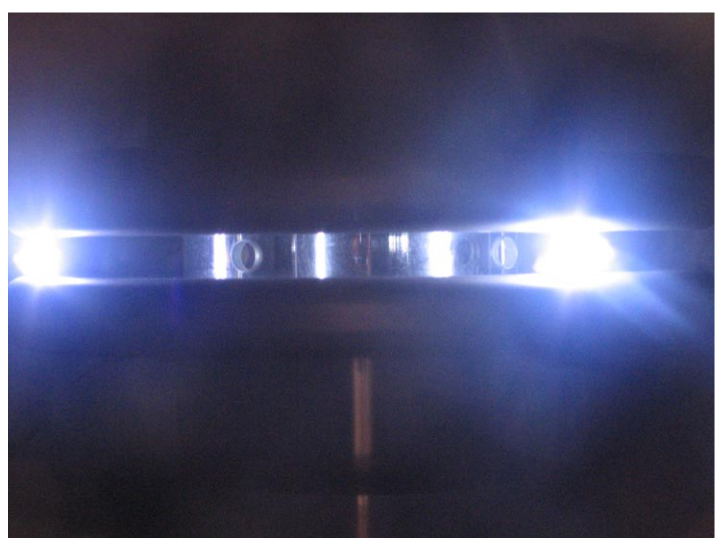


Photo of multichannel breakdown in air (P=1 atmosphere).

- Completed design and drawing package for a 1.5 MV switch
- Project directed toward smaller scale multi-channel physics experiments
- Designed small test stand
- Constructed test stand
- Started preliminary tests

Significance

With the completion of the design of the 1.5 MV switch, most of the electrical and mechanical design issues have been considered and dealt with. The design demonstrates that it will be possible to build such a switch, and key design issues are known. The small scale tests now under way will characterize multichannel switch behavior as a function of voltage pulse rise time and electrode diameter. With this information it will be possible to design a full-size 6 MV switch with greater confidence and with a good idea of what to expect for performance (i.e., how much lower the switch inductance should be expected to be).

Development of Simulation and Validation Techniques for the Dynamic Behavior of Metals at the Grain Scale

93530

T. J. Vogler, E. B. Marin, W. M. Trott, C. S. Alexander, D. J. Bammann, C. J. Kimmer

Project Purpose

The behavior of metals during high strain rate and shock loading as related to the material microstructure has not yet been fully addressed. Such studies require modeling techniques based on multiscale approaches that quantitatively represent the underlying microstructure and physical processes that are operative in these loading regimes. Through this project we are developing a validated modeling capability for dynamic deformation of metals subjected to high strain rate, high-pressure loading, using a grain-level representation of the microstructure.

The mesoscale approach uses both an extended crystal plasticity model to represent the most relevant physics of high strain rate-induced deformation, and three-dimensional (3D) finite element models with embedded realistic grain structures. Such modeling has only become possible with the most recent generation of advanced supercomputing machines. Model development is supported by state-of-the-art experimental equipment (line-VISAR) to generate grain-scale dynamics data for model validation. At present, the line-VISAR (velocity interferometer system for any reflector) is likely the only diagnostic tool able to probe the grain-level response under dynamic loading, though its full potential for research of this type has not yet been exploited.

This approach, which brings together current computational capabilities and newly developed grain-scale diagnostic tools, will produce a methodology for validating grain-level material models for applications in the high strain rate and shock loading regime. Most importantly, it will help to build an understanding of outstanding issues regarding the strength of materials under shock and ramp loading, knowledge needed to address many critical material dynamics response problems.

In addition, the model framework will also be applicable to characterize the material properties and performance of system components whose characteristic dimensions are comparable to the grain size, such as microelectromechanical systems (MEMS). This project is focused on tantalum because of DOE interest in it as a body-centered cubic, high-density metal, as well as because of other modeling work being done with it.

FY 2006 Accomplishments

During the initial part of this project, our work on the modeling side focused on extending the crystal plasticity formulation to the high strain rate, high-pressure regime. First, we formulated the framework in an explicit manner to be consistent with the anticipated high-rate problems. High strain rate effects have been accounted for by the incorporation of phonon drag effects into the kinetics of slip.

The formulation for including pressure in the crystal plasticity framework is in development. We demonstrated the functionality of the current model in 3D simulations of a Taylor impact cylinder for regimes similar to those of interest for the plate impact experiments of this project. Finally, initial work is under way for implementation of the model into Alegra.

We performed experimental work in three areas. First, we conducted an experiment on single crystal tantalum with (100) and (110) orientations on Z. These results will provide important information about the high-pressure behavior of tantalum crystals for calibration of the model, something that did not previously exist. Second, we attempted a line-VISAR experiment with polycrystalline tantalum on Z but, due to experimental difficulties, obtained no usable data. It appears that line-VISAR is not a functional capability on Z. Finally, we are preparing experiments with the line-VISAR on tantalum for the gas gun and small pulser. This involves special sample preparation as well as ensuring that the line-VISAR system is fully operational.

Significance

The work of this project is important in at least four ways. First, and most importantly, it will provide the basis for using crystal plasticity to address issues of dynamic material behavior. Crystal plasticity has been used extensively in the static regime but hardly at all in the high-rate regime. One area where this should prove valuable is for parts where the grain scale is similar to the part size (e.g., MEMS). Crystal plasticity should provide a means for modeling that is more predictive than current continuum models. Another area this could impact is the design of parts for weapon systems so that the microstructure (grain size and orientation) can impact their dynamic behavior in a desired manner.

Second, this project should impact the entire multiscale modeling approach for the dynamic regime. A true multiscale modeling approach should consist of models for the relevant size/time regimes (e.g., atomistic, lattice, meso-, and continuum scales) that are linked together so that key physics behavior is passed up in scale.

One point that is sometimes forgotten, though, is that each of these individual models must be validated against experimental data. To date, no comprehensive means for validating dynamic simulations at the mesoscale has been developed, largely because of the difficulty in obtaining experimental data. This project should provide that, which should ultimately affect modeling not only of polycrystalline metals but also of energetics, porous materials (thermal batteries, etc.), and heterogeneous materials (e.g., alumina-filled epoxy). This project, therefore, will be an important step in developing a predictive modeling capability for dynamic material behavior.

Third, it should provide insight into certain very fundamental issues that have remained unresolved by the shock physics community. In particular, it should help us to better understand the state of a polycrystal following passage of shock wave and answer questions such as how long does it take to reach equilibrium, how important is the microstructure of the material, and how will it respond when perturbed from that state?

Finally, it will lead to a better understanding of the dynamic behavior of tantalum, a material of significant DOE interest.

Dynamic Compression of Synthetic Diamond Windows

93531
D. H. Dolan III, J. Podsednik, T. J. Vogler, M. D. Knudson

Project Purpose

Windows are an integral part of dynamic compression research but often limit the range of experiments that can be performed. To extend our experimental capabilities, we are studying synthetic diamond as a window for shock and isentropic compression research. Diamond is of interest because it is transparent over a broad spectral range, has a large elastic limit, and is extremely stiff. Traditionally, diamond has been far too expensive to use in single-event experiments, but this is changing as diamond synthesis technology matures.

The first objective for this project is to characterize the behavior of single-crystal diamond windows under shock compression. The key challenge is to determine the stress range where diamond is elastic and transparent. Next, we will turn to isentropic compression measurements, where it is believed that diamond may be driven to even higher stresses while continuing to function as a window. The final stage of this project will be to apply synthetic diamond to a problem such as the study of liquid isentropes.

Synthetic diamond production is not nearly as well established as other types of windows (e.g., sapphire), so the material is somewhat difficult to acquire and does not currently have the consistency normally expected of an optical window. However, the long-term benefits of synthetic diamond windows are immense.

Within the dynamic compression field, diamond opens up a new range of experiments that can be performed, such as high-pressure liquid isentrope studies, visible/infrared pyrometry, and dynamic strength studies. Furthermore, there is great interest in combining static and dynamic compression, in essence creating a "disposable" diamond anvil cell.

Investment in synthetic diamond technology has broader benefits to Sandia and DOE, ranging from microelectronics/micromechanics to novel detection systems for use in pulsed power environments.

FY 2006 Accomplishments

Much of our efforts in FY 2006 were directed at obtaining a consistent supply of synthetic diamond windows. We received several diamond shipments from our collaborators at the Carnegie DOE Alliance Center (CDAC). After several iterations, CDAC refined their growth and polishing protocols to provide suitable diamond windows. We also placed an order with Apollo Diamond, and began receiving windows in May 2006. Windows from both sources will be tested to determine if there is a substantial difference in the elastic behavior. Impact experiments are scheduled in the second quarter of FY 2007.

In preparation for these experiments, a portable fast VISAR (velocity interferometer system for any reflector) was constructed to obtain the best possible time resolution. This system should provide subnanosecond resolution and avoid ambiguities noted in previous Sandia studies of natural diamond. While designing this system, we developed a new type of VISAR geometry, which allows extremely low fringe constants in a small cavity space.

We developed analysis for the diamond experiments by studying elastic and inelastic compression of shocked cubic zirconia. For symmetric impact geometry, inelastic deformation may be subtle in the initial compression state. However, there are secondary features that reveal inelastic behavior: VISAR contrast loss, transparency loss upon unloading, and multiple wave structure in the free surface release. These findings will be important during the interpretation of upcoming diamond experiments.

We measured an unexpected VISAR correction for cubic zirconia, which proved to useful in the analysis of previous experiments on the Z machine.

Significance

Synthetic diamond opens up a wide range of experimental possibilities in high pressure research. Diamond may be useful as a high-impedance window in shock compression experiments up to 1 Mbar, and perhaps even further in isentropic compression. This capability will broaden the kinds of experiments that can be performed on the Sandia Z/ZR. Applications include static precompression (i.e., squeezing a sample to an initial stress prior to shock or isentropic compression), liquid isentrope research (such as deuterium or water), and material strength studies. Diamond is also used in high-speed x-ray bolometers and neutron detectors at Z, and may lead to novel diagnostics for pulsed power and inertial confinement fusion research.

By characterizing synthetic diamond windows in shock and isentropic compression experiments, we provide a capability that can be used in many other settings. Diamond has many applications as a robust optical/infrared window, but the expense of natural diamonds limits its use. Our research will stimulate the development of synthetic diamond production for technical applications, and may help reduce material costs in future work.

There are numerous applications for synthetic diamond if the material becomes readily available. Diamond is a semiconducting material and can be doped to create electronic devices. Such devices could benefit from the extreme hardness of diamond and its substantial thermal conductivity (a concern in future high-speed microprocessors). Diamond is biocompatible and has a very low frictional coefficient, which may have impacts in microelectromechanical systems (MEMS).

Other Communications

D.H. Dolan, “Applications of Diamond in Dynamic Compression Research,” presented at New Ideas Research Forum, Sandia National Laboratories, Albuquerque, NM, March 2006.

Fast High-Voltage Spark Gap Switch with a Phase-Changing Dielectric

93532
K. W. Reed, G. L. Brennecka, F. E. White, S. F. Glover, J. E. Martin

Project Purpose

High-voltage (HV) switching is central to pulsed power systems, so a lot of ongoing research and development is focused on improving switch performance. A survey of recent work shows that the focus is directed at improving one or more critical parameters, with recent emphasis on reliability, low triggered jitter (allows synchronization), fast primary switches (essential for modularity), and the ability to operate repetitively. Efforts to understand and promote multichanneling for lower on-state inductance and increased switch longevity are under way. With a focus on reliability and repetitive operation, much work is being done to improve solid-state devices to a level that allows their use in pulsed power applications.

A rarely used technology that meets all of the criteria for pulsed power systems is the solid dielectric switch. These switches have found applications in single shot, single switch machines since the early 1970s, but their use has fallen off since the beginning of the new millennium as pulsed power development began to focus more on switches rated for repetitive use and/or large scale systems with high switch counts.

Solid dielectric switches are characteristically compact compared to alternative technologies and have been demonstrated with various combinations of multi-MA current capacity, several hundred kV DC hold off, low-on-state inductance (1 nHy was demonstrated in a 5 MA electrically triggered switch), < 1 mΩ on-state resistance (typical), low switching loss, and extremely fast switching due to the negligible resistive phase. Solid dielectric switches reliably self-trigger or they can be command triggered explosively, electrically, and conceivably by using a small solid-state laser, since any photoelectrons would rapidly avalanche in the highly over-volted solid – though no record of laser triggering has been found. The primary disadvantage is that the dielectric must be replaced after each firing.

The purpose of this research is to develop solid dielectric materials that can be reused, allowing solid dielectric switches to be used repeatedly or in large numbers without replacement between shots.

FY 2006 Accomplishments

Hardware Design and Assembly

A sealed test cell has been designed and assembled that contains the sample material between specially designed electrodes. During testing, the cell is housed in a vacuum cross that immerses it in an envelope of electrically insulating gas (nitrogen or SF_6) when high voltage is applied. The test cell is electrically connected to the HV power supply via a coiled wire inside the field grading cup. The negative electrode and test cell assembly is connected to ground through a calibrated 1 kΩ current viewing resistor (CVR) allowing the current into that electrode to be monitored on an oscilloscope. Corona currents are captured by the grounded vacuum cross and bypass the CVR.

Solid dielectric testing methods in ASTM D 3755 and 149 provide a standard for comparison of relative material dielectric strengths. Conversely, our goal is to test materials in switch geometries with representative electrode spacings, over as large an area as permissible. This drives the use of a Rogowski electrode profile corresponding to a 1 mm electrode spacing yielding a maximum uniform field of 1000 kV/cm with an applied voltage of 100 kV. The specially engineered materials we are developing are completely novel, one-off formulations making sample materials one of our biggest costs and driving the decision to limit sample size to 25 mL. Consistent with these constraints, the diameter of the uniform field region between electrodes is 1 cm.

Test voltage from a Glassman HV power supply is controlled by an arbitrary function generator. Expediting a basic understanding of the behavior of these materials, initial test voltages will ramp linearly to 100 kV in 30 seconds – Z MARX's charge to 90 kV in about two minutes. As material behavior is better understood, and as optimal materials are identified and developed, the application of test voltage will be modified to better represent that of Z. A calibrated Spellman voltage divider is used to monitor the actual voltage being applied to the test cell. ES&H (environment, safety, and health) documentation and the readiness review are completed. Initial testing is under way.

Materials Technology Development
We completed baseline tests of commercially available materials. The most significant potential contribution of this research to the HV switch technology is in the application of Sandia's world class expertise in materials science to the development of special materials. We made progress in developing engineering tools to tailor materials to the application. We tested the first engineered material and two more engineered materials will be delivered and tested very soon.

Significance

The DOE Pulsed Power Initiatives serve a broad spectrum of mission objectives. These include controlled fusion, stockpile stewardship, testing materials, and electronic components' response to radiation, characterizing equation-of-state for critical materials, detecting underground threats, materials processing and surface morphology, sterilization of foods and hazardous materials, code validation and the study of electromagnetic and radiation-hydrodynamics.

HV switches are the only active component in all pulsed power systems used by DOE to accomplish these missions. The switching speed, inductance, voltage-hold-off, losses, jitter, and reliability of these switches dominate the maintenance requirements, costs, compactness, reliability, and ultimate performance of the pulsed power systems they control.

A solid-dielectric switch provides optimal performance in all of these critical parameters but requires replacement of the broken dielectric between every shot. Many present, and particularly future, pulsed power requirements for DOE missions necessitate either/both repetitive operation or large-scale integration of switched components. Presently, the high performance capability of solid dielectric switches cannot be applied to DOE pulsed power needs because it isn't feasible to replace dielectrics between shots on a repetitively pulsed machine or on one with large numbers of switches.

Success of this project would eliminate the problem of quickly and repetitively restoring the solid dielectric in these high performance switches and make solid dielectric switching a viable design option in future DOE pulsed power systems.

Development of a Physics Understanding of Pulsed Power Closing Switches for Multiple LTD Applications

93533
J. R. Woodworth, D. L. Johnson, J. A. Alexander, J. J. Leckbee, G. S. Sarkisov, M. G. Mazarakis, B. V. Oliver, K. R. Prestwich

Project Purpose

Linear transformer drivers (LTDs) are a developing pulsed power architecture that may dramatically lower the cost and size of many future pulsed power accelerators. The performance of present linear transformer drivers, however, is limited by both the reliability and the relatively high inductance of the many high-pressure gas switches used in these devices.

We are performing this research to gain a solid physics understanding of the operation of present gas switches used in LTDs and to develop improved gas switches with lower inductance and high reliability. The present switch design has about 100 nanohenrys of inductance. Decreasing the switch inductance to ~ 50 nanohenrys would allow design of classes of LTDs with much faster pulse rise-times and higher efficiencies than present systems.

FY 2006 Accomplishments

We set up a switch testing facility in a new laboratory and began testing the baseline switch design currently used in LTD accelerators. We are also constructing an automated switch lifetime testing facility for use with future switch designs. The baseline switch is pressurized with dry air and has six each 6-mm gaps in series. Twenty nanofarad capacitors on the top and bottom of the switch are direct current-charged to + or – 100 kV, respectively, and then a high-voltage pulse is applied to the middle electrode of the switch to trigger the switch.

Our initial results using electrical diagnostics and a fast optical camera indicate that the trigger pulse will break down one or two gaps in the switch, but there can be as much as a 100 ns delay between the breakdown of the first two gaps and the breakdown of the entire switch. Individual 6-mm-wide gaps appear to break down in a time that is fast compared to the 5 ns time resolution of our framing camera. We are setting up a spectroscopy system to study the light emitted by the switch breakdown arcs versus time. This will tell us how the electron density and temperature of the arcs develop during the breakdown process.

We designed and are fabricating two competing low-inductance switch designs that may lower the overall switch inductance by a factor of two, from its present ~ 100 nanohenry value to ~ 50 nanohenrys.

Significance

Our initial results suggest that the operation of the linear transformer driver switches is critically dependent on the details of the trigger pulse in ways that had not been understood previously. Also, the very rapid formation of individual breakdown arcs in the switches (less than 5 ns) suggests that there is room for significant improvements in switch technology.

High-Current Carbon Nanotube Electron Sources

93534
P. A. Miller, M. P. Siegal, T. R. Lockner

Project Purpose

The purpose of the project was to develop arrays of carbon nanotubes that could be used as cathode material for long-pulse intense relativistic electron beam accelerators. Our goal was cathode material capable of emitting 10 amperes of electron current at greater than 1 ampere per square centimeter for 1 second with an applied acceleration potential of 50 kilovolts.

FY 2006 Accomplishments

In the first part of the year, we developed a test bed with a diagnostic suite and a computer control and data-acquisition code for measuring electron emission from carbon nanotube samples. We fabricated arrays of nanotubes in a variety of geometries on silicon wafer substrates. We tested emission from several samples and obtained detailed emission data.

In order to improve the conductivity of the substrate upon which we deposited the nanotubes, we developed a process for coating the silicon wafers with conducting tungsten layers. During the balance of the year, we developed and tested carbon nanotubes fabricated under a variety of conditions. None gave good electron emission. Consequently, the project was discontinued.

Significance

We learned more than we had known before about the relationship of various processing conditions to the resulting characteristics of nanotube arrays fabricated by chemical vapor deposition. We also learned that the approaches we investigated did not result in superior electron emission characteristics.

Precision Electron Flow Measurements in a Disk Transmission Line

93535
M. E. Savage, T. D. Pointon, C. W. Mendel Jr.

Project Purpose

The purpose of the project is to understand electron flow in low-impedance, high-current magnetically insulated transmission lines (MITLs) and verify the numerical modeling of those systems. Because the electron sheath thicknesses are a small fraction of the vacuum gap width, it is difficult to model these systems due to the small calculation cell sizes required. In addition, the small energy of the flowing electrons makes numerical noise energy significant, and electron emission thresholds have a significant effect. Our experimental goals are to make careful measurements of electron flow in a high-current disk MITL and to develop a probe that can measure electron flow directly.

Normally, electron flow is measured by subtracting cathode current from anode current, which has large uncertainties when the difference is small compared to the anode current. A direct measurement of electron flow, which has not been done before, would allow measurement of electron flow on large drivers such as Z or Saturn. Direct electron current measurements on a large driver would allow verification of the accuracy of assumptions used in the design of those systems.

It is important to understand the electron flow in those systems because electron flow and inductance are competing requirements in our present design strategy. Inductance essentially sets the efficiency, and therefore the cost, of large z-pinch drivers. In order to optimize performance of large drivers, it is essential to minimize the inductance of the final transmission lines. An intelligent optimization requires a detailed understanding, and corroboration with relevant experiments. Our experiments will involve two different MITL designs, both with careful conventional and novel electron flow diagnostics. Numerical simulations of the design and comparison of the predictions to the experimental data are important parts of the project.

FY 2006 Accomplishments

We developed a simple analytic model for transmission line design based on the assumptions that the electron flow is most important early in the drive pulse, that the z-pinch load does not change inductance significantly early in the pulse, and that the transmission line electromagnetic wave transit time is short compared to the pulse length. We neglected plasma gap closure initially, although it could easily be added later. The model allows calculation of the transmission line impedance profile (as a function of radius) analytically. The model accepts a parameter that is the desired change in electron flow current with distance.

Since the ratio of load inductance to time is less than the transmission line vacuum impedance early in the pulse, the voltage across the transmission line decreases toward the load. The transfer of electrostatic energy to magnetic energy allows betatron acceleration of electrons; the resulting electron energy increase allows electrons to return to the cathode in some cases. In addition, the slowing electron drift velocity due to the falling voltage allows the electron sheath to develop large electric fields.

Electrons can reach the anode or return to the cathode in some cases due to those space-charge fields. Simulations show that electron flow tends to occur at a value determined by analytic theory, unless geometric changes are too abrupt. It is possible to prescribe the change in equilibrium flow in the model and to calculate a transmission line impedance profile that meets the chosen electron current loss rate.

The experimental hardware has been designed using the model, and most of the parts have been procured.

Significance

Sandia has as one of its missions development of pulsed power knowledge and expertise. This project is intended to increase knowledge in an important subject area for the delivery of large electromagnetic energies (currents) to physically small loads. This is the essence of high energy density physics.The experiment will allow refinement of the numerical models for high-current drivers. Accurate models are important for optimal (efficient) designs. Development of the direct electron current measurement diagnostic would allow measurement of electron flow on a large high-current driver, further increasing the accuracy and relevance of the models.

LTD/RTL Power Flow Development for Z-Pinch Fusion Drivers

101817

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Project Purpose

Z-pinch inertial fusion energy (Z-IFE) complements and extends the single-shot z-pinch fusion program on Z to a repetitive, high-yield, power plant scenario that can be used to produce electricity and hydrogen, transmute nuclear waste, desalinate water, and so on, with no CO_2 production and no long-lived radioactive nuclear waste. Earlier research addressed critical issues and led to acceptance of Z-IFE by the broader fusion community. The Z-IFE concept uses a linear transformer driver (LTD) accelerator and a recyclable transmission line (RTL) to connect the LTD driver to a high-yield fusion target inside a thick-liquid-wall power plant chamber.

The purpose of this project was to investigate power flow issues in the LTD driver, the intermediate adder/transmission lines, and the RTL. All of these issues are also applicable to a single-shot inertial confinement fusion (ICF) high-yield fusion facility, as is envisioned by Sandia. This research is vital to producing a credible and cost-effective path to high yield and IFE.

FY 2006 Accomplishments

We investigated the key physics issue for RTLs: the power flow at the high linear current densities that occur near the target (up to 5 MA/cm). Effects include surface heating, melting, ablation, plasma formation, electron flow, magnetic insulation, conductivity changes, magnetic field diffusion changes, possible ion flow, and RTL mass motion. We studied these effects theoretically, computationally (with the ALEGRA and LSP codes), and experimentally (at the Kurchatov Institute, Moscow). All of the results show that RTL power flow at high linear current densities (up to 7 MA/cm in experiments) should work for the RTL concept.

We also investigated voltage adding and repetitive operation with LTD cavities. We addressed repetitive switching operation, electrode erosion effects, and heating effects due to the high average power. A 0.5 MA LTD cavity was operated in a repetitive mode at Sandia, with gas purging between shots, in order to demonstrate automated operation at rates of 25 seconds or less between shots. After over 3,000 shots were fired, the last 50 shots were fired once every 10.3 seconds (much better than the original goal, and essentially meeting the desired goal of 10 seconds for a Z-IFE power plant). At the High Current Electronics Institute in Tomsk, Russia, five LTD 1.0 MA, 100 kV cavities were successfully combined into a voltage adder configuration with a test load to study the system operation at 1.0 MA, 500 kV.

The complete power flow includes the LTD driver, the intermediate adder/transmission lines, and the RTL. We used particle-in-cell codes to study the electron flow losses produced near the magnetic nulls that occur when coaxial LTD lines are added together. We found that higher currents, with fewer lines, lead to less electron loss. Circuit model codes were used to model the complete power flow circuit. We proposed LTD architectures for drivers at 60 MA (at Sandia) and at 90 MA (at Kurchatov) for high yield and for IFE.

Results from all of these power flow studies greatly help to validate the LTD/RTL concept for single-shot ICF high yield and for repetitive-shot IFE.

Significance

Our research enables development of a credible, cost-effective approach to fusion high yield and to Z-IFE. The high-yield goal supports the NNSA goals for ICF for stockpile stewardship. The IFE goal supports Department of Energy goals for energy security for the nation through unlimited fusion energy.

This project holds high potential to lead to a long-range fusion energy future for Sandia over the next several decades. The potential benefit is much broader than just the pulsed power sciences area and would extend throughout Sandia.

Z-Pinch-Driven Fusion Systems for IFE, Transmutation, and GNEP

102362
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Project Purpose

In this six-month Z-Inertial Fusion Energy (Z-IFE) project, we made an effort to create a logical path to reach the ultimate goal of a fusion power plant. To illustrate this path forward, a roadmap was created outlining the current issues, needed steps, and time required to reach these goals. Past Z-IFE research concentrated mainly on the full-scale plant design.

The proposed z-proof of principle (Z-Pop) facility was conceived to address some of the needed science and engineering development required before the full-scale power plant could be built. However, some of the fundamental physical understanding and experimentation needed before building such a facility had not been addressed.

The purpose of the roadmap was to gain an understanding of what it would take to develop Z-IFE. It begins with using existing facilities capable of handling various separate experiments. These facilities include Sandia's Z-Beamlet and ZR, where several containment and mitigation experiments can be conducted. The next step is to build an engineering test facility (ZN) where automation, recyclable transmission line (RTL), target fabrication, and driver development can take place and eventually be integrated.

With the inception of the Global Nuclear Energy Partnership (GNEP) initiative and the goal of a closed fuel cycle, an alternative use for z-pinch-driven fusion arose. Work conducted through the Grand Challenge LDRD project "Advanced Fusion Concepts: Neutrons for Testing and Energy" showed that fusion neutrons can transmute the actinides in spent fuel. The ZN facility will also be the test bed for developing z-pinch inertial confinement fusion transmutation. Once everything is integrated, ZN will become a pilot plant that will be the final stage before the full-scale transmutation and power plants are built.

The Grand Challenge LDRD project concentrated on developing the early steps in the roadmap involving containment experiments on ZR. Our Z-IFE progress extends the work completed for that project, focusing on the full-scale transmutation and power plants.

FY 2006 Accomplishments

We made substantial progress in the areas of shock mitigation, chamber design, RTL development, flibe (F, Li, Be) chemistry, and systems cost models for both the transmutation and power plants.

Our transmutation work focused on designing a chamber capable of using the high-energy neutrons from a z-pinch reaction to burn actinides. The key advantages to transmuting actinides with this design are:

- a safer design than seen with faster reactors
- no additional actinides, since no fertile material such as uranium is required
- valuable operating experience that will be needed for the full-scale power plant.

A new, higher repetitive-rate thick liquid curtain chamber design was proposed based on the results of the systems cost analysis. This proposed chamber design requires a slightly larger target yield and more than fives times the repetitive rate of the original Z-IFE design.

Modeling results of aerosols-absorbing x-rays show promise that they may be able to mitigate fusion yields up to 3 GJ. Based on these results, a new first-wall chamber design was conceived that may significantly reduce the complications associated with the thick liquid curtain designs.

We created a systems cost model to optimize the number of chambers, the target yield, and shot rate needed for a fusion power plant. The results indicated that fewer chambers at a higher repetitive rate and yield are more economical than the original Z-IFE power plant design.

A detailed cost analysis of the linear transformer driver (LTD), the device that supplies the power to the fusion target, was conducted for the power plant facility. The results indicated that it will be a significant percentage of the total cost of the plants.

We conducted various studies of the chemical interaction between a flibe-like salt and a steel RTL post shot. This was to gain an understanding of how the free ions of the dissociated salt and steel interact. Further experimentation on the electrical properties of a frozen flibe RTL was conducted to understand its effectiveness of transmitting the pulse to the target.

We analyzed four power conversion systems: supercritical CO_2 cycle, Rankine cycle, Brayton cycle, and a combined cycle. Assuming that high-temperature materials are available in the future, the analysis showed that the combined cycle is best for both transmutation and power plants.

We completed a tritium permeation study of the piping and chamber and compared the results to an analysis conducted for the International Thermonuclear Experimental Reactor (ITER). The results showed that using a permeation barrier coating on the pipes along with optimizing partial pressure and diameter could reduce tritium leakage significantly.

Significance

The ZR-machine provides an ample facility for conducting the necessary physics and engineering experiments required for increasing fusion yield, demonstrating fusion containment, and target development.

Sandia's talented robotics group will be able to aid in designing the automation required for maneuvering the RTLs in and out of the containment chamber. Sandia experts in materials will be valuable in several aspects of this work, as material development for the vessel, RTL, and power plant will be essential to ensure safe operating conditions. Sandia programs support energy research and development ranging from solar and wind to nuclear power. There are also several years of research completed through the Z-IFE program on using z-pinch ICF to create commercial power. Recently the focus of Z-IFE has shifted to include using z-pinch fusion for transmutation, tying Z-IFE directly into the GNEP initiative and its focus on a closed nuclear fuel cycle.

Sandia can take the lead in developing z-pinch driven ICF energy for both power production and transmutation.

Quasispherical Direct-Drive Fusion

103459
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Project Purpose

This work was performed to evaluate the feasibility of quasispherical direct-drive (QSDD) fusion on machines such as ZR at 28 MA and 150 ns, and on larger and/or faster machines.

FY 2006 Accomplishments

We studied one technique for current sharpening, the magnetically insulated current amplifier (MICA), and made an engineering design. The purpose of the MICA is to sharpen the current pulse delivered by ZR to a quasispherical load. With a faster pulse, quasispherical shells implode faster, are less susceptible to instabilities, and are more likely to drive deuterium-tritium (DT) to ignition.

Two-dimensional (2D) simulations of quasispherical implosions have shown that on slower machines with implosion times greater than 100 ns it may be possible to use a double-shell system to ignite an inner shell filled with DT. In the double-shell system, the outer shell implodes onto a layer of DT that generates plasma pressure, which in turn implodes an inner gold shell filled with DT. On ZR simulations, the inner shell has ignited with a 12 MJ yield, but in this system there is very little margin for ignition. A double-shell system for a 56 MA driver at 150 ns implosion has produced a simulated yield of 130 MJ with considerable margin in attaining the necessary temperature and density-radius product for ignition.

In the double-shell systems considered, a wire basket rather than a complete shell forms the outer shell that accelerates to velocities over 20 cm/µs. At slower velocities the inner-shell fuel will not reach the 2 keV ignition temperature required for ignition in a system where bremsstrahlung radiation trapping is strong. For such systems as ZR with 150 ns implosion times, it will not be possible to fabricate single complete shells because they would be too thin. For faster systems with implosion times less than 100 ns it may be possible to directly drive a single shell to volumetric ignition. The mass of DT that can be heated to ignition in a single-shell system is about four times the mass that can be heated to ignition in the inner shell of a double-shell system.

One way to realize a faster system is through the use of MICA. In MICA, the vacuum section to the feed is replaced by spiraling magnetically insulated transmission lines. Vacuum closing switches atop the transmission lines couple the current to the quasispherical pinch. Using such a system, the normal 150 ns, 28 MA pulse on ZR can be converted to a 40 MA pulse with a 30 ns rise time.

Upon completing an engineering study of a MICA system for ZR, we confirmed that this fast MICA pulse can implode a single quasispherical shell. One-dimensional (1D) simulations of faster single-shell implosions on ZR with a MICA have shown yields of over 100 MJ. Yields with 2D simulations are expected to be lower. We are currently performing 2D simulations of single-shell implosions faster than 100 ns.

Significance

Direct-drive ignition on ZR or a slightly larger machine will impact several applications, including the production of a pulsed neutron source, a neutron source for fission power plant waste transmutation, and fusion energy.

S&T Strategic Objectives

Superhydrophobic Surface Coatings for Microfluidics and MEMS

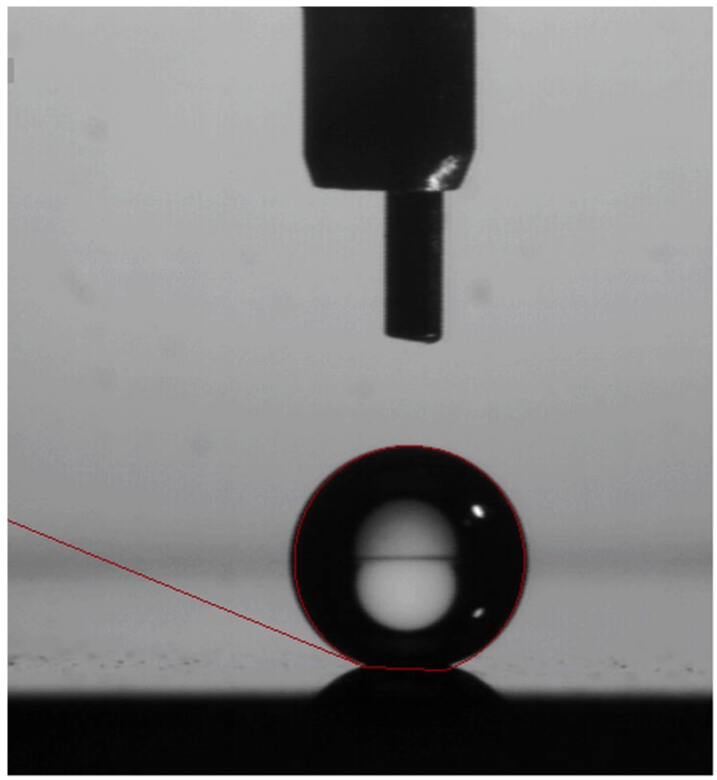
73185

F. B. van Swol, T. L. Sounart, J. E. Houston, S. Singh

Project Purpose

The purpose of this project was to develop a fundamental understanding of the conditions that cause liquid droplets to roll rather than flow/slide on a surface and how this "rolling transition" influences the boundary condition describing fluid flow in a pipe or microchannel. Rolling of droplets is important for aerosol collection strategies because it allows trapped particles to be concentrated and transported in liquid droplets with no need for a predefined/micromachined fluidic architecture.

We also studied the superhydrophobic (SH) water interface and interactions between submerged SH surfaces. In general, the water/SH interface is important to understanding and achieving water exclusion (e.g., to prevent corrosion or avoid stress corrosion cracking). Interactions between submerged SH surfaces are important in understanding colloid stability and protein folding.



Water droplet on superhydrophobic silica surface

FY 2006 Accomplishments

We used an interfacial force microscope to examine synthetic aerogel-like SH films (with measured contact angles of greater than 165º and a contact angle hysteresis between 2-5º) underwater. We discovered a very long-range hydrophobic attraction that was due to out-of-contact evaporation, or cavitation, of the intervening water at tip-to-substrate separations ranging from 0.8 µm to as much as 3.5 µm. Cavitation is a first-order phase transition characterized by a sudden, strong attractive force and by the appearance of a vapor bridge spanning the tip-to-substrate gap.

Preexisting nanobubbles of a size commensurate with the interaction length have been proposed as a source of long-range interactions. We therefore used in situ confocal imaging to search for bubbles on an isolated, flat SH surface. Neither the microscopy nor neutron reflectivity experiments provided evidence for bubbles – certainly not micrometer-sized bubbles. We therefore argue that cavitation is a consequence of, and thermodynamically consistent with, the properties of confined water. The critical separation, D, below which cavitation is thermodynamically favored, can be estimated from Laplace's equation as 1.4 µm: this is a lower bound and will increase if Δp, the pressure difference across the interface, is reduced by incorporation of air into the cavity.

We completed an experimental study of a low-Reynolds number shear flow between two surfaces, one of which had a regular grooved texture augmented with a superhydrophobic coating. The combination reduces the effective fluid-surface contact area, thereby appreciably decreasing the drag on the surface and effectively changing the macroscopic boundary condition on the surface from no slip to limited slip. We measured the force on the surface and the velocity field in the immediate vicinity on the surface (and thus the wall shear) simultaneously. The latter facilitated a direct assessment of the effective slip length associated with the drag reduction.

Significance

Long-range attractive forces have been observed for more than 20 years, and different theories have been proposed to explain the origin of these forces. Our experiments reveal that cavitation can be one source of long-range hydrophobic interactions. For rough surfaces, the interaction length extends from the nanometer scale to the micrometer scale. Correspondingly, interaction lengths of hundreds of nanometers, observed for smooth surfaces (for example, snap-to-contact), might also be explained by cavitation, in agreement with thermodynamic expectations. These long-range forces, especially cavitation, might play an important role in protein folding phenomena and many other diverse phenomena that occur under water.

Refereed Communications

S. Singh, J. Houston, F. van Swol, C.J. Brinker, "Superhydrophobicity - Drying Transition of Confined Water," *Nature*, vol. 442, p. 526, August 2006.

S.R. Challa and F. van Swol, "Molecular Simulations of Lubrication and Solvation Forces, "*Physical Review E*, vol. 73, p. 016306, January 2006.

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S.J. Kwoun, R. Cairncross, R.M. Lec, C.J. Brinker, and P. Shah, "The Study of Interaction of Superhydrophobic (SH) Materials with Fluids Using TSM Sensors," in *Proceedings of the IEEE International Frequency Control Symposium and Exposition*, pp. 1400-1403, August 2005.

Other Communications

C.J. Brinker, "Surfactant-Directed and Living Cell-Directed Assembly of Porous and Composite 3D Nanostructures," presented (invited) at Universite Pierre et Marie Curie, Paris, France, June 2006.

C.J. Brinker, "Bridging the Gap: Observation of Ultralong-Range Hydrophobic Interactions," presented at the American Chemical Society 232nd National Meeting, San Francisco, CA, September 2006.

F. van Swol and S. Challa, "On the Interplay Between Hydrodynamic and Solvation Interactions," presented at Pacifichem, Honolulu, HI, December 2005.

S. Challa and F. van Swol, "The Interplay of Solvation Forces with Lubrication Forces in Thin Gaps," presented at the 58th Annual Meeting of the APS Division of Fluid Dynamics, Chicago, IL, November 2005.

F. van Swol and S. Challa, "Molecular Simulations of Lubrication and Solvation Forces," presented at ASCR/LANL, Los Alamos, NM, August 2005.

F. van Swol, "Superhydrophobic Surfaces: Bouncing and Rolling Droplets," presented at Pacifichem, Honolulu, HI, December 2005.

C.J. Brinker, "Evaporation Induced Self-Assembly of Porous and Composite Nanostructures," presented at Rutgers University, Distinguished Alumnus Award Address, New Brunswick, NJ, March 2006.

S. Challa and F. van Swol, "Influence of Solvation Forces on Material Characterization," presented at the Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2005.

C.J. Brinker, "Surfactant-Directed and Living Cell-Directed Assembly of Porous and Composite 3D Nanostructures," presented (invited) at St. Gobain Céramiques Avancées Desmarquest, Montreuil, France, June 2006.

C.J. Brinker, "Symbiotic Assembly of Bio/Nano Interfaces and Architectures," presented (invited) at Northwestern University, Evanston, IL, September 2006.

Achieving a New Paradigm in Software Technology

79883
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Project Purpose

Large software systems are very costly and manpower-intensive to build and even more expensive to modify and maintain. Those systems that are actually delivered (industry surveys indicate that one third of large software development projects are abandoned without completion) are typically far behind schedule, greatly exceed projected costs, and are essentially riddled with flaws (bugs) that can cause unexpected failures. Surveys also indicate that, since the mid-1990s, US industry has lost between $60-90 billion each year due to abandonment or repair of software projects.

Our economy and national security have become increasingly dependent on costly and unreliable software programs. Software has become, in effect, a stealth US infrastructure. Yet, the enormous costs have driven US industry to off-shore the development of this crucial infrastructure to sensitive countries including India, China, and Russia.

This project is an ambitious effort to help put an end to these serious and long-standing problems. We seek to revolutionize how software systems are created, used, and modified for both US industry and government agencies using a new approach called self-assembling software. Our patented self-assembling software technology is inspired by the robust biological self-assembly processes carried out by dynamic protein networks in living cells.

Our goal is to significantly expand the novel capabilities of our self-assembling software approach, to bring this differentiating capability to the point where it can have substantial impact on Sandia and DOE programs, and to dramatically alter the course of a trillion-dollar world-wide industry. Our success in implementing this technology will create a "disruptive" Sandia technology with enormous potential impact and economic value.

FY 2006 Accomplishments

In FY 2006 we began implementing the self-assembling software environment that we designed and evaluated the previous year. The environment consists of:

- a real-time compiler written in Pentium 4 assembly language
- a powerful set of generic building blocks that can self-assemble to perform general computing tasks yet circumvent the need for tedious and error-prone programming using conventional programming language syntax (e.g. C++, JAVA)
- infrastructure to customize building blocks to self-assemble in the proper functional structures in response to user guidance
- a novel and intuitive graphical user interface that is also built from self-assembling components
- an auto-journaling system that automatically saves and provides searchable access to literally everything done by a user in the environment (verb executions, data accesses, even navigation).

Specific accomplishments are:

1. Ported our completed Pentium 4 compiler to personal computers (PCs) with FreeBSD, Windows/Cygwin, and QNX operating systems
2. Implemented building blocks and the infrastructure to customize their self-assembly
3. Implemented much of the graphical interface with novel self-assembling interface components that both generate OpenGL screen graphics directly and interact with the other self-assembling code building blocks. Using both SDL and OpenGL makes our interface portable to multiple operating systems.

Approximately 40,000 lines of working code have gone into implementing items (2) and (3). Approximately 100,000 lines of working assembly code have gone into (1).

We are also working to protect the intellectual property (IP) we generated. We submitted 13 technical advances on the self-assembling building block and interface design innovations developed for this environment and submitted two patent applications.

Significance

High costs, poor reliability, and difficulties in modifying large software packages create vulnerabilities for our national security and the US economy. Our national security and military effectiveness are becoming increasingly dependent on large software systems, and the high costs and unreliable performance of such systems put our national security at risk. Further, commercial software development is increasingly outsourced for development in sensitive countries. The software that US industry uses from these sources could also be introducing serious vulnerabilities into our nation's infrastructures.

Our implementation accomplishments provide great confidence that our new approach to software development is sound. The implementation of our approach will in turn produce a "disruptive" technology for cost-effective and reliable software development like no other currently available. This technology could be used to address the key economic and national security issues listed above. For example, within the DOE alone, our techniques could provide a new generation of large software tools (e.g., alarm systems monitoring software analogous to Argus, security vulnerability simulation tools analogous to JCATS) with greater reliability but without costing the many tens of millions of dollars that DOE and the Department of Defense have invested to build and maintain the existing systems.

The successful development and commercialization of this disruptive IP could have significant and widespread benefits to the nation's economy and national security, and Sandia IP ownership and licensing of this revolutionary technology might provide unprecedented future royalties to Sandia. Our long-term vision includes a revitalized US software industry that can afford to hire US citizens to produce reliable software for our nation.

Robust Spore-Based Detection System

84270
T. W. Lane, J. N. Kaiser

Project Purpose

Cell-based detectors have the potential for exquisite selectivity and sensitivity; however, the challenges associated with keeping most cells viable in a detector system are profound. The ultimate goal of this project is to create a robust cell-based detection system using signal transduction processes found in bacterial spores.

Some bacteria produce spores that are remarkably resistant to extremes of temperature and desiccation and are stable for years without refrigeration. *Bacillus subtilis* spores germinate to form vegetative cells upon addition of specific chemical germinants. Upon binding of the germinant to individual receptors on the spore, the first step in germination is release of dipicolinic acid.

The purpose of this project was to alter the signaling process through genetic engineering to create chimeric germinant receptors that are specific for individual biothreat agents such as toxins. When the specific agent binds to the receptor (single binding event), it will elicit the release of many molecules of dipicolinic acid, constituting an amplified signal that can be readily detected. Genetically engineered spores with different specificities can be used singly or in combination to create detectors for a range of biothreat agents. The first key step (proof-of-principal) is the creation of receptors with altered specificity.

Since the spores will remain dormant until activated by the specific analyte/germinant, this system will be stable over a range of temperatures, resistant to desiccation and will not require growth medium to maintain the viability of the biological detector system. Thus, it will have clear advantages over other cell-based detection systems.

FY 2006 Accomplishments

In previous work we developed a colony lift-based germination assay that appeared to be amenable to high throughput analysis methods. Initially this assay, which was critical to the success of the project, appeared to function in a consistent and reproducible manner. Early this year the assay stopped functioning in the same manner, which constituted a major roadblock to further development efforts. We spent significant time troubleshooting the assay and developing alternatives. We developed two different liquid-based assays as replacements, but they proved to be insufficient for high-throughput applications.

The second technical road block arose when we attempted to use the cloned germination (Ger) operon to complement Ger deletion mutation strains. While it appeared that we were able to restore the germination activity of the mutant strains, the level of activity that we were able to achieve was not as high as that of the wild-type strain and not sufficiently above background for an assay to be successful.

We were not able to overcome these technical hurdles. As a result, we deemed this approach for developing spore-based detector systems to be too fraught with technical hurdles for further pursuit.

Significance

Sandia has an important mission to develop detectors for chemical and biological agents (pathogens and toxins) for war fighters and first responders. These detectors need to be sensitive, rapid, and accurate in order to provide a detect-to-warn capability. In addition, detectors must be stable enough to endure long-term storage and provide a long operational lifetime in the field. Cell-based biosensors are good candidates for providing the necessary sensitivity, speed, and accuracy, but to date these systems are prone to contamination, are sensitive to environmental conditions, and have a short operational life.

A successful spore-based detector will combine the sensitivity of a cell-based biosensor with the stability required for a fieldable device. It can be stored dry, in a shipping container, in the desert, for years until needed. Once activated the spore-based biosensor does not require growth media and so is resistant to bacterial contamination.

This research has added to the base of knowledge about the mechanism of germination in *Bacillus subtilis* and *Bacillus cereus*. Germination is a crucial first step in the disease pathway in the related organism *Bacillus anthracis*. Although we will not be working with *Bacillus anthracis,* what we learn in this research could lead to a greater understanding of this select agent.

This research aligns well with the future directions of both the Biotechnology and the Homeland Security investment areas. In this work, we are developing the supporting technology for a next-generation sensor system architecture. Future work in developing the sensor architecture will lie at the interface between biotechnology and engineering.

PCSS/Fiber-Optic Trigger System for Pulsed Power Switches

86362

S. F. Glover, K. W. Reed, F. J. Zutavern

Project Purpose

This project will develop an optically controlled solid-state trigger generator (TG) for high-voltage switches (HVSs). The photoconductive semiconductor switch (PCSS) is an enabling technology for this objective. PCSS-based TGs provide a number of advancements in trigger generation. Subnanosecond rise time and jitter will enhance HVS performance over existing triggering techniques. Fiber optic control of a PCSS-based TG physically located on an HVS will allow the triggering hardware to float, thereby eliminating the need for high-voltage cables traversing high potential fields or for line-of-sight-optics.

Fiber optically isolated triggers can be independently controlled, monitored, and adjusted from a central control room, resulting in improved performance and reliability. Using PCSS-based TGs, instead of existing trigger systems that require high-voltage cables or line-of-sight optics, results in greater simplicity in] design, and reduces space and maintenance requirements. High-speed system diagnostics will also be improved by the reduced jitter of the HVSs, allowing more precise measurements.

Proof-of-concept demonstration has resulted in a 300 kV gas switch delivering 5 kA to a 50 ohm load with 100 ps jitter using a PCSS-based TG delivering a 40 kV trigger pulse with 500 ps rise time. However, significant research is still necessary in order to make this technology practical. This project will address: (1) direct current (DC) and pulsed charging HVS for conventional and waveform shaping systems, (2) PCSS protection with an intermediary switch, (3) direct triggering of HVS by PCSS, and (4) a scalable design for a large range of voltage and current applications.

FY 2006 Accomplishments

We used a PCSS-based TG to trigger DC-charged trigatron, rimfire, and radial pin style switches. The last of the testing with trigatron style switches used a PerkinElmer commercial switch. The rms (root-mean-square) jitter of the PCSS output current and the triga-tron output current were measured as low as 100 ps and 1.2 ns, respectively. The 100 ps PCSS rms jitter reinforces past PCSS triggering performance obtained when triggering a 300 kV hydrogen gap trigatron switch.

Testing continued with the PCSS-trigatron circuit by using the trigatron output to trigger a 200 kV rimfire switch designed for linear transformer drivers (LTDs), resulting in 30 ns rms jitter. Placing the trigatron between the PCSS and the rimfire switches provided higher fan-out capabilities since it could handle larger currents than the PCSS without significantly impacting lifetime. Also, a higher-voltage trigger pulse was attainable since the trigatron could operate at 100 kV and the available PCSS support circuitry could not.

Up to this point, PCSS had only been used to directly trigger trigatron style switches. The next step was to directly trigger a midplane style switch. Initial work involving the ±50 kV radial pin switch, (RPS) designed by Kinetech (operated at ±30 kV), demonstrated the potential for subnanosecond jitter with small data sets. However, further optimization and testing are necessary.

We improved the high-bandwidth diagnostics in the new circuit, continued jitter analysis, continued determining the minimum PCSS TG requirements to trigger the RPS with low jitter, and investigated how the PCSS is affected by transients from the main switch (in this case the RPS). This research will provide additional insight into the performance of the PCSS and the level of protection it requires in preparation for optimizing the compactness of the PCSS-based trigger system and designing an integrated switch/trigger system in FY 2007.

Significance

HVSs are the basis of all pulsed power systems used by the DOE in the areas of pulsed power and high energy density sciences and military technologies and applications. HVSs are a key element in determining system reliability, maintenance, cost, compactness, and optimal performance. Laser trigger systems are expensive and require sophisticated optics. Therefore, a reliable trigger system using state-of-the-art technology is essential to supporting DOE missions.

HVSs are the basis of all pulsed power systems used in DOE by

- Energy: for controlled fusion
- Defense Programs: for stockpile stewardship, testing materials and electronic components response to radiation, detecting underground threats, code validation, and the study of electromagnetic and radiation-hydrodynamics
- Office of Science: for characterizing equation-of-state for critical materials and materials processing and surface morphology.

Other Communications

S.F. Glover, F.J. Zutavern, K.W. Reed, M.E. Swalby, A. Mar, M.L. Horry, F.E. White, and F.R. Gruner, "Fiber-Optic Controlled PCSS Triggers for High-Voltage Pulsed Power Switches," in *Proceedings of the 2006 International Power Modulator Conference*, May 2006.

Nanostructured Surfaces for Microfluidics and Sensing Applications

91991
N. S. Bell, S. T. Picraux, M. Piech, D. Yang

Project Purpose

The purpose of this research is to investigate the design and synthesis of nanowire surfaces with unique properties for manipulating fluids and sensing chemicals. Advances in the emerging fields of nanotechnology and microfluidics are creating new opportunities for ultrasensitive molecular analysis in lab-on-a-chip systems based on electronic transport through semiconducting nanowires. However, fluids become exceedingly difficult to manipulate at the very small micro- and nanoliter volumes of interest.

Also, viable methods to position nanowire sensing elements between large arrays of electrodes are needed to exploit these structures for ultrasensitive chemical sensing in small fluid volumes. Thus, new methods are necessary both to manipulate small fluid droplets on surfaces without excessive sticking and to organize nanoscale structures on surfaces for electronic sensing in liquids. The first objective of this project is to understand and control light-induced fluid droplet motion on surfaces for liquid manipulation and sensing. The second objective is to demonstrate methods for ex situ electrical doping of silicon nanowires and their alignment between electrode arrays.

FY 2006 Accomplishments

We developed a method to form photoswitchable azobenzene monolayers tethered to the surface of silicon chips and demonstrated the ability to move liquid droplets on this surface using only light. In our design the azobenzene molecules are covalently bound to the oxidized Si surface through an aminopropylmethyldiethoxysilane linkage that allows the azobenzene to be reversibly switched between its *cis* and *trans* states, respectively, by exposure to visible and ultraviolet light. These two states have significantly different surface energies, allowing spatially dependent light-induced switching of the interaction strength between liquids and the surface.

We completed a systematic study of the light-induced surface interaction for a variety of fluids (water, diiodomethane, 1-bromonaphthalene, 1-methylnaphthalene, dimethylformamide, acetonitrile, benzonitrile, formamide, ethylene glycol) to characterize and better understand the roll of the surface chemistry in this method of controlling liquid droplets on surfaces.

We established the criteria for fluid motion: the photoinduced change in surface contact angle for the liquid must exceed the fluid-surface interaction hysteresis (advancing minus receding contact angles) under visible illumination. For the representative fluids for differing types of surface chemical interactions, we achieved contact angle photoswitching from 8 to 15 degrees and demonstrated the light-controlled motion on smooth surfaces for several of the fluids meeting this criteria, including benzonitrile and 1-bromonaphthalene.

Our results provide the scientific foundation for incorporating these controlled surface switchable monolayers onto our patterned Si surfaces, which contain micropillars and nanowires to enhance the surface hydrophobicity. This advance will enable light-controlled manipulation of a wide variety of liquid droplets on nanostructured surfaces.

In the second part of this project, we developed a method for ex situ doping of Si nanowires and demonstrated the positioning and contacting of the nanowires between prepatterned electrodes on oxidized Si substrates. Our new ex situ doping method for nanowires was accomplished in a two-step proximity doping approach using a spin-on dopant (a boron containing glass). The process involves a short thermal treatment with the spin-on source in proximity to the Si nanowires followed by a second drive-in diffusion.

We used the approach for three different dopant concentrations in the 10^{17}-10^{19}/cm^3 range, and we characterized the resulting nanowires structurally and electrically using scanning electron microscopy, transmission electron microscopy, secondary ion mass spectrometry, and I-V (current-voltage) transport. We aligned the nanowires between Au electrode arrays spaced 2 and 4 µm apart by ultrasonically placing the electrically doped nanowires into solution and positioning them between electrodes by dielectrophoretic alignment. We made low-resistance electrical contacts to the nanowires by patterned deposition and are optimizing the processing steps to minimize the resistance and maximize the sensitivity of the nanowires for chemical sensing.

Significance

Advances in the emerging fields of nanotechnology and microfluidics are converging to create new opportunities for ultrasensitive molecular analysis in lab-on-a-chip systems based on electronic transport through semiconducting nanowires. However, several advances are needed to exploit these opportunities. First, fluids become exceedingly difficult to manipulate at the very small microliter to nanoliter volumes of interest. Also, methods to efficiently position nanowire sensing elements between electrodes and electrically contact them must be developed to exploit these structures for ultrasensitive chemical sensing in small fluid volumes.

This project has demonstrated a new method of using only light to manipulate nanoliter fluid droplets on surfaces without excessive sticking. We developed a method based on design of monolayer coatings of tethered azobenzene molecules that can be reversibly switched using only light. This design, in combination with our earlier demonstration of surface nanostructuring to achieve low-hysteresis superhydrophobic surfaces, offers a new approach to manipulating fluid droplets in a digital approach for microfluidic analysis.

A second key requirement for exploiting nanotechnology for ultrasensitive chemical sensing through electronic transport in nanowires is the electrical doping and organizing of the nanowires between electrodes on surfaces. For those cases where electrical doping of the nanowires after growth is required, we have developed a doping technique derived from Si technology and successfully extended it to the small dimensions of these highly sensitive nanoscale structures. By demonstrating the dielectrophoretic alignment of these nanowires between 20 element electrode arrays and demonstrating a contacting method for these nanowires, we have shown the key steps necessary for creating electronic nanowire chemical sensors.

Responding to the Identified Gap and National Needs in Early Bioresponse

93536

K. W. Boyack, V. M. Peck, G. S. Davidson

Project Purpose

This project focuses on the concept and field of syndromic surveillance, or the detection of disease states within a population using surveillance of various data types prior to confirmed diagnosis by clinical testing. The purpose of this project was to begin to address the gap in syndromic surveillance: a need for better data types and algorithms to improve (decrease) detection times for diseases within a population.

FY 2006 Accomplishments

At the outset of the project, we performed an extensive survey of the surveillance literature. This survey identified existing systems and their various strengths, along with the need for better validation of surveillance systems and algorithms. We found that there has been no clear validation of any surveillance system, no concurrent comparison between competing systems on the same data types and populations. Thus, there is no way to say how good syndromic surveillance is.

We also attempted to acquire both bovine and human data that would allow us to begin independent testing of algorithms and detection methods. However, these attempts were unsuccessful due to privacy and other concerns.

We next met with experts in New Mexico who had both interest and expertise in syndromic surveillance and epidemiology, who wanted to be involved in addressing the gap in disease surveillance, and who had connections (which we at Sandia did not have) in the public health community, both regionally and nationally. We participated in the Research Association of Medical and Biological Organizations (RAMBO), a community of public health researchers in the state, and presented our findings, both to small groups of people from RAMBO and to the group as a whole. Most members of the group agreed with the identified gap and needs. Our findings confirmed and codified the gap that many members of the group had suspected was there.

We formed a team including key people from the state (Dr. Gary Simpson, Dr. David Broudy), as well as practitioners from hospital systems (including Waco, TX, and St. Louis, MO), and came to a consensus on a future research direction. To that end, we documented descriptions of current surveillance systems, identified limitations, and recommended a direction for a large-scale validation study involving multiple surveillance systems, data types, and communities. A key facet of this direction is that multiple systems must be tested in multiple communities simultaneously. Such a study has never been done.

Significance

A large scale validation study of syndromic surveillance will constitute a significant contribution to public health. Sandia would participate in such a study as an honest broker. It is not our place to replace the health care institutions in their roles, but we can add great value to the process as an independent arbiter of data and analysis and by performing a meta-analysis that we expect to lead to a system of systems. This would be an appropriate role for Sandia due to our expertise in data mining, algorithms, and systems analysis using large-scale data.

Sandia will be in a position to contribute to a problem of national significance – the detection of disease states and potential epidemics (e.g., bird flu) that could pose a threat not only to Sandia's ability to meet its mission needs, but also to the health of the country as a whole.

Viral Vectors for Gene Modification of Plants as Chem/Bio Sensors

93537

S. M. Brozik, D. C. Arango, M. Manginell, P. L. Dolan

Project Purpose

Chemical or biological sensors that are specific, sensitive, and robust are sorely needed for intelligence gathering for verification of nuclear nonproliferation treaty compliance and detouring production of weapons of mass destruction. Although much progress has been made in the area of biosensors, improvements in sensor lifetime, robustness, and device packaging are required before these devices become widely used.

Current chemical and biological detection and identification techniques require sample collection from the field followed by transport to a laboratory for analysis. In addition to being expensive and time consuming, results can often be inconclusive due to compromised sample integrity during collection and transport. We propose to fill this need by altering established, mature plants into chem/bio sensors.

Our goal is to design a safe, noninfectious vector that can be used to invade, replicate, and introduce foreign genes into mature host plants that then allow the plant to sense chem/bio agents. The genes introduced through the vector included a reporter gene that encodes for green fluorescent protein (GFP) and a gene that encodes for a mammalian receptor that recognizes a chemical agent. In this proof of concept study, GFP was induced by the presence of 17b-estradiol (estrogen). Detection of fluorescence indicates the presence of the target chemical agent.

Because the sensor is a plant, costly device packaging development or manufacturing of the sensor are not required. Additionally, the biological recognition and reporting elements are maintained in a living, natural environment and therefore do not suffer from lifetime disadvantages typical of most biosensing platforms. Detection of the chem/bio agent reporter (GFP) can be accomplished discretely as detection is only at a specific wavelength.

FY 2006 Accomplishments

Our FY 2006 milestones were aimed at designing a safe, noninfectious vector which will be used to invade, replicate, and introduce foreign genes into mature host plants that then allow the plant to sense chem/bio agents. We were able to demonstrate these goals in a seedling plant. We used *Agrobacterium* species mediated transformation to introduce the plasmids into the plants. We demonstrated the feasibility of using inoculated plants as chemical sensors by exposing the plant to varying concentrations of estrogen via moisture in the soil. The timed response of the plant to the target analyte was monitored by measuring green fluorescence of the leaves upon exposure to an ultraviolet (UV) lamp and laser excitation under a light microscope.

Transformation of *Agrobacterium* strain EHA105 with the *pER-GFP* vector

We used spectinomycin selection and *Agrobacterium* transformation kit from Qbiogene, Inc. The plasmid, *pER8-GFP* (developed at Rockefeller University) was used to transform *Arabidopsis thaliana* host plants. The *Arabidopsis thaliana pER8-GFP* is a transgenic plant with an estrogen receptor which expresses GFP reporter protein in response to 17b-estradiol.

Plant growth and seed harvesting

Plants were grown in a growth chamber supplied by LEHLE Seeds and were sown in styrofoam cups growing 1-3 plants/cup. The first bolts were clipped as soon as they appeared to promote the growth of inflorescences.

Agrobacterium-mediated transformation of *Arabidopsis*

The *Agrobacterium* were grown to mid-log (OD600 ~ 2). A few days before transformation *Agrobacterium* was grown in YEP or LB plus spectinomycin (110 μg/mL) at 28 ºC, 200 rpm. The cells were harvested by centrifugation at 5500 g for 20 minutes at room temperature. Prior to use they were resuspended in infiltration (dipping) medium to a final OD600 of 0.08 to 2.0.

Floral dip and harvesting
Plants with inflorescences of about 5 cm were selected for the first inoculation. The *Agrobacterium* inoculum was applied drop-by-drop to all closed flower buds and this process was continued over the course of a couple of weeks. Once the first silique started to brown, we discontinued watering. Seeds were harvested after the siliques completely browned and stored in small envelopes at room temperature. To select transformants, an antibiotic marker was used.

Induce expression of GFP using 17b-estradiol
Seeds were either grown in the presence of 17b-estradiol or plants were grown on noninductive medium for 2-3 weeks then transferred to inductive media. Verification of GFP was obtained by Northern analysis and fluorescence microscopy.

Significance

A critical mission of Sandia is to provide innovative technology to solve any problem that threatens the peace, freedom, and security of our nation. In line with that mission, our technology promises an innovative approach towards detecting and detouring production of nuclear, chemical, biological, or conventional weapon agents that threaten the peace and security of America and its allies while establishing Sandia as the leader in chem/bio sensing. The plant-based sensors described in this work are an innovative solution to this important goal with substantial potential payoff. If successful, this technology will be of high impact to several Sandia investment areas including Nonproliferation and Assessments and Biotechnology. If successful, this work will yield a revolutionary method for altering established, mature plants into chem/bio sensors. These sensors provide valuable solutions to the major strategic challenges of gathering viable intelligence regarding nuclear nonproliferation treaty compliance and production of chemical, biological, or conventional warfare agents.

LIGA-Fabricated Composite Right/Left-Handed Metamaterials

93538
M. A. Forman, D. R. Boehme, R. J. Punnoose

Project Purpose

The purpose of this project is to understand how high-impedance surfaces comprised of composite right/left-handed metamaterials can be used to improve antennas for laboratory applications. Some weapons and communications applications, for instance, require the placement of omnidirectional antennas near a metallic surface. Normally, a metallic surface in close proximity to an antenna would have detrimental effects on its input impedance and radiation pattern. However, a high-impedance surface, comprised of a sheet patterned metal and dielectric, placed over the metallic surface will allow the antenna to function normally.

Additionally, high-impedance surfaces have the ability to convert rectangular waveguides, a transverse electric (TE) and transverse magnetic (TM) guiding structure with a cutoff frequency, into a transverse electromagnetic mode (TEM) waveguide with no cutoff frequency. A metamaterial-loaded waveguide thus has the potential of carrying high power, while being much smaller than their solid metal counterparts. Finally, low-volume metamaterial-loaded horn antennas have the potential of replacing bulkier antennas in existing systems. This could prove useful in retrofitting older systems with additional antennas for enhanced functionality.

FY 2006 Accomplishments

We fabricated multiple sheets of composite right/left-handed metamaterial to serve as a high-impedance surface for use in antenna applications. We measured the input impedance and far-field patterns of several omnidirectional antennas in proximity to the metamaterial. Relative to a placement near a metallic surface, the antennas in close proximity to the metamaterial had significantly improved input impedances and radiation patterns. With some antennas, the loading provided by the metamaterial even improved the input impedance over that of an antenna with no loading.

Additionally, we fabricated nine horn antennas with varying geometries using horizontal aluminum conductors and vertical metamaterial sheets. We measured the input impedance and patterns of the antennas for all geometries and found that metamaterial-loaded horn antennas carry a TEM mode and thus have no cut-off frequency. A single horn antenna with an aperture of 17.5 x 17.5 mm, a fraction of the size of a standard 2.5 GHz antenna, functioned well (input impedance, bandwidth, and pattern) at 2.5 GHz. The horn antennas were also tested with and without metamaterial backplanes to determine the effect on the horn feed location.

Significance

Composite right/left-handed metamaterials permit omnidirectional antennas to be placed in close proximity to metallic surfaces. This has immediate weapons and sensor system applications allowing deployed or integrated communications systems to be placed close to metallic surfaces (weapon bodies). Metamaterial-loaded waveguides and antennas have the potential of carrying high power, while being much smaller than their solid-metal counterparts. This could prove useful in retrofitting existing systems with additional antennas for enhanced functionality.

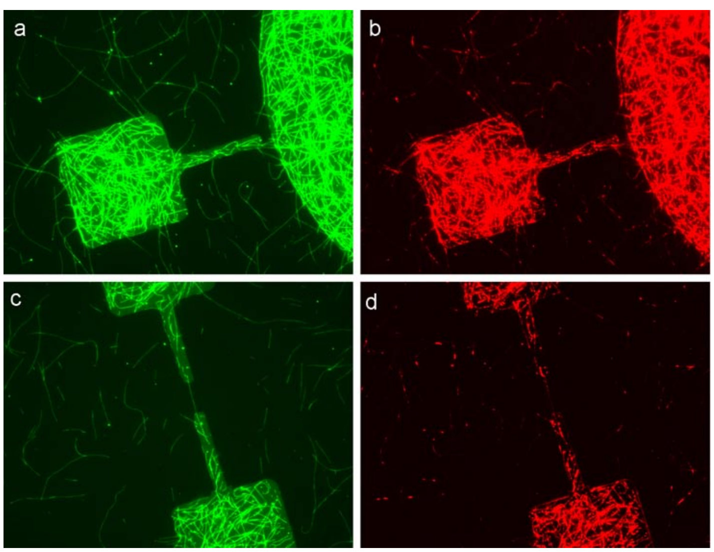
Active Assembly for Large-Scale Manufacturing of Integrated Nanoelectronics

93539
E. D. Spoerke, C. M. Matzke, B. C. Bunker, G. D. Bachand

Project Purpose

The integration and organization of nanostructures into functional devices is one of the most significant challenges impeding the widespread application of nanotechnology. Although methods for nanoparticle assembly such as flow-induced patterning and nano-manipulation have been explored, these methods lack control and cannot be practiced on a large scale.

The purpose of this project is to develop an innovative approach to nanostructure manufacturing using biomolecular machinery as tools to assemble nano-devices. These tools, microtubules and motor proteins, may be manipulated through careful control of their specific biochemistries and selective, cooperative interactions with one another.



(a, c) Colored micrographs showing fluorescently labeled microtubules (MTs) bound selectively to patterened gold structures. MTs are visible bridging between the different structures as a cluster in (a) and as a single MT in (c).

(b, d) Colored micrographs showing fluorescently labeled carbon nanotubes bound to the microtubules in (a, c).

The strong a/b, c/d correlation in fluorescence illustrates the templating of the carbon nanotubes onto the selectively captured MTs. The square pads are approximately 25 microns across.

Following nature's example, we have created an organized microtubule network that serves as a templating scaffold to organize nanomaterials onto lithographic platforms. This approach represents a unique and potentially powerful method to organize and assemble a variety of nanomaterials, with potential applications including microelectronics, packaging, and sensor development.

FY 2006 Accomplishments

We demonstrated the bioselective self-organization of microtubule (MT) networks onto lithographically patterned substrates and the subsequent application of these organized microtubule networks for nanocargo assembly. This process has involved a number of developmental stages.

1. Selective chemical functionalization of lithographic gold patterns: We used a discriminatory chemical treatment to create a chemical "blocking layer" on silica surfaces between lithographically-patterned gold features. As opposed to using a bulk blocking agent, such as a photoresist, this approach took advantage of the orthogonal surface chemistries of the gold and silica. This more specific approach allowed the process to be tailored more specifically for the chemical and biological systems of interest.

2. Specific biofunctionalization of lithographic patterns: We incubated a buffered solution of motor proteins with the selectively functionalized surfaces, effectively decorating only the "unblocked" gold surfaces with active kinesin motors. Because of the engineered selectivity of the attachment and the solution-phase nature of this process, the treatment is applicable even to large-scale systems.

3. Bioselective Attachment of Microtubules to Lithographic Patterns: We captured polymerized microtubules out of a liquid suspension by the motor proteins adhered to the patterned gold substrates.

This process relies on the very specific, cooperative interactions between the microtubules and the kinesin motors. The kinesin motors, chemically activated in nature by the hydrolysis of adenosine triphosphate to adenosine diphosphate have adapted to attach to and translate along microtubules. By sequentially releasing and reattaching to the MT with different segments of the protein, the motors effectively “walk” along the MT.

We modified this behavior by treating the motors with adenylylimidodiphosphate, a nonhydrolyzable analog to adenosine triphosphate. Under these conditions, the motors may attach to microtubules but cannot release them as they normally would during motor motility. The subsequent binding produced a very high microtubule capture rate, preferentially on the kinesin-coated gold patterns. These microtubules were found not only attached to gold surfaces, but depending on MT length, bridging designed gaps between these electrically conductive gold contacts.

4. Use of Microtubule Networks to Assemble Nanocargo: We used specific biochemical ligands to attach nanocargo, such as gold nanoparticles and carbon nanotubes, along the microtubule networks. Taking advantage of strong, bioselective linking agents, such as biotin and streptavidin, we were able to bind functionalized carbon nanotubes or gold nanoparticles on the captured, organized MT scaffolds.

This assembly motif generated nanoscale structures where carbon nanotubes or gold nanoparticles were positioned and assembled to form microtubule-templated bridges between the designed structures of the patterned gold.

Significance

The development of these capabilities is significant to a number of organizations both internal and external to Sandia. Internally, this work has benefited other existing research projects, including another LDRD project (Large Scale Manufacturing of Integrated Nanostructures for Sensing) as well as a Basic Energy Sciences-funded biointegration project on active assembly. It has also contributed to an active Center for Integrated Nanotechnologies (CINT) collaboration with Professor Robert Haddon at the University of California-Riverside.

On a more general note, this project addresses one of the chief limitations of nanomaterials progress – directed assembly. This research has helped to establish a new technical capability and to promote Sandia’s national nanomaterials leadership role. With significant opportunity to impact fields such as microelectronics, manufacturing and packaging, sensing technologies, and microsystem integration, this work is relevant to programs within Sandia, to research at CINT, and to the nanomaterials community at large.

Refereed Communications

E.D. Spoerke, G.D. Bachand, A.M. Trent, B.C. Bunker, J. Liu, C.E. Warrender, A.M. Bouchard, and G. Osbourn, “Biologically Directed Nanoscale Materials Assembly,” presented at the Materials Research Society Spring Conference, San Francisco, CA, April 2006.

Molecular Electronics: Theory and Experiment

93540
S. V. Faleev, D. Robinson

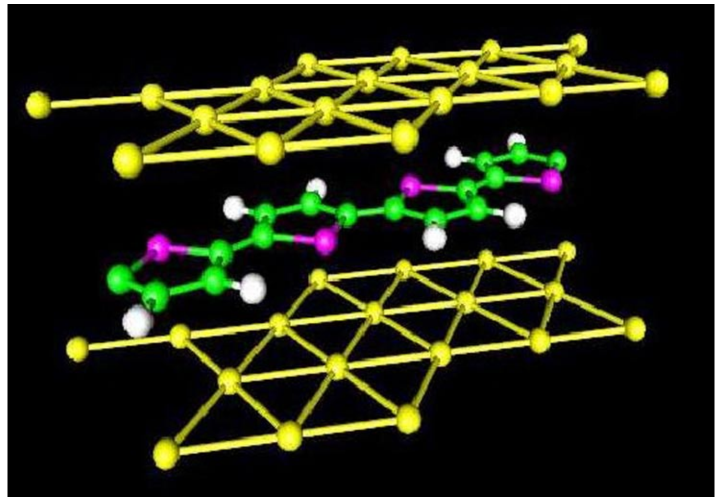
Project Purpose

The purpose of this project is to extend a first-principles state-of-the-art computational method developed recently at Sandia for simulation of the electronic transport through nanosystems attached to metal electrodes with applied voltage bias [1] to describe tens to hundreds of atoms in active region.

By nature, first-principles quantum-mechanical approaches are necessary in order to theoretically describe the properties of a single molecule or similar size nanosystem attached to electrodes, because quantum effects dominate the transport characteristics of such nanoscale systems. The first-principles electronic structure methods available today are highly developed and sophisticated but are implemented mostly to describe systems that are in equilibrium and either periodic or have a finite number of atoms. However, nanoscale systems connected to bulk electrodes should be described by methods that can treat nonperiodic and infinite size open systems. Additionally, in the presence of finite voltage bias applied across the electrodes, the system is out of equilibrium, and the theoretical methods should properly describe this nonequilibrium situation.

Our method is based on the local density approximation of density functional theory and is implemented in the framework of the linear muffin-tin orbital approach in its atomic sphere approximation. A fully atomistic description of the electrodes and the nanosystem is used, and the self-consistent charge and electrostatic potential for the system under applied bias is calculated using the nonequilibrium Green's function (NEGF) approach. This is the first such ab initio NEGF method that does not use the pseudopotential approximation for description of core electrons.

The goal of the experimental side of the project was to develop a new platform for molecular electronics experiments that is more scalable and more well-defined than existing approaches, and make transport measurements on model systems that were comparable to theoretical calculations.



Geometry of single layer of polythiophene between electrodes.

FY 2006 Accomplishments

We extended the existing first-principles computational method for simulation of the electronic transport through nanosystems attached to metal electrodes in order to describe systems with tens to several hundreds atoms in active region. Previously this approach has been applied to less than 15 atoms in the active region.

We applied our first-principles computational method to calculate conductance of pi-stacked thiophene polymers sandwiched between Au(111) contacts. The active region in our simulations consisted of 1 to 8 polythiophene layers arranged parallel to the plane of the Au(111) surface (corresponding to from 14 to 112 atoms in the active region). We found that the transmission coefficient for electrons on Fermi surface is rather large for such systems, slowly reducing from 0.3 to 0.1 when the number of layers increases from 2 to 8. The fact that the transmission coefficient decreases with increasing number of layers rather slowly (not exponentially) can be explained by finite density of states at Fermi energy for the system of pi-stacked thiophene polymers; thus transmission is not dominated by a tunneling mechanism.

We developed a simple and elegant open-face molecular electronics platform that is easy to fabricate and can be interrogated electrically and spectroscopically at the same time. To prepare samples, we dip-coat a photolithographically defined interdigitated array of gold electrodes on an oxidized silicon wafer with gold or silver nanoparticles coated with an insulating surfactant. We make background measurements and then exchange the insulating molecule for a more conducting one by a simple solvent soak, and repeat measurements. The spectroscopic method takes advantage of the surface enhancement of Raman spectra that occurs when the molecules are between small metal particles.

We made measurements that demonstrate dramatic conductance increases when molecules are exchanged, and showed spectroscopically that applied voltages must be bounded to avoid degradation of the molecules. We obtained results that are comparable to the theoretical results reported in the earlier work [1].

We developed a platform intended for study of mercury-contacted vertical device structures using block copolymer-patterned substrates. While our platform is promising for this application, it was more valuable for the study of conducting polymers and nanowire growth, topics of two other LDRD projects.

Significance

Recent experimental developments have made it possible to fabricate electronic devices where the active part is a molecule, a nanotube, or a similar structure of nanometer-scale dimension. These developments have given rise to growing interest in theoretical methods that can calculate the electronic structure and transport properties of nanoscale devices from first principles.

In this work we extended our existing first-principles computational method to study systems with several tens to several hundreds of atoms in the active region. This extension significantly widened the range of possible applications of the method. In this project we applied our method to calculate electronic structure and transport properties of conducting polymers (specifically, pi-stacked thiophene polymers).

This is an interesting system because conducting polymers hold great promise for radiation detection and solar energy conversion and are used in electronics and illumination. We found that the transmission coefficient of pi-stacked thiophene polymers arranged in planes parallel to gold electrodes shows slow nonexponential decrease of conductance with increasing number of molecular layers.

The experimental methods developed in this work extend the state of the art of device fabrication in this field. The ability to use an independent, in situ spectroscopic characterization technique while making measurements will add confidence to current understanding of the physical and chemical processes that occur when current is passed through molecular layers. The block copolymer patterning technique has enabled us to make measurements that have made significant contributions to our understanding of charge transport through organic polymers.

[1] S.V. Faleev, F. Léonard, D.A. Stewart, and M. van Schilfgaarde, "Ab Initio Tight-Binding LMTO Method for Nonequilibrium Electron Transport in Nanosystems," Physical Review B, vol. 71, p. 195422, May 2005.

Refereed Communications

D.B. Robinson and A.A. Talin, "Nanolily Pads for Study of Short-Range Electron Transport," presented at the MRS spring meeting, San Francisco, CA, May 2006.

Design and Synthesis of Tailored Multidimensional Nanoscale Structures

93541

J. L. Lee, S. K. Griffiths

Project Purpose

A key to the widespread application of nanomaterials lies in the ability to customize their shape, size, and composition as desired. Among the many potential fields of application for tailored nanostructures are biomedicine, nanoelectronics, sensors, optics, microfluidics, and microelectromechanical systems (MEMS), many of which are of interest to Sandia's national security programs. Although growth of zero- and one-dimensional (0D, 1D) nanostructures of a variety of materials has been demonstrated, the synthesis of tailored two- and three-dimensional (2D, 3D) nanostructures is an underexplored area.

The purpose of this project is to demonstrate an expanded range of shapes, sizes, and compositions of tailored multidimensional nanostructures that can be created in order to increase the flexibility and functionality of nanomaterials.

To address the objective of creating nanomaterials with specific shapes, sizes, and compositions, we have partnered with Professor Zhong Lin Wang and his postdoctoral associate Puxian Gao, of the Georgia Institute of Technology, who have extensive experience in synthesizing novel nanoscale structures. We proposed customizing the shapes of 2D and 3D nanostructures of various materials by forming them from the intersections of multiple 1D and 2D nanostructures.

We grew the lower-dimensional (1D, 2D) nanostructures on the faces of tailored 3D substrates using a vapor phase technique, where the substrate shape allows them to grow in such a way that they intersect to form higher-dimensional (2D, 3D) nanostructures.

An example of a substrate shape that may allow for the growth and intersection of lower-dimensional nanostructures are substrates having an L-shaped cross-section. These substrates were created using the nanoimprint lithography (NIL) tool at Sandia and will be subjected to the controlled introduction of surface defects to investigate their effect as catalytic sites for nanostructure growth. Several methods are proposed for introducing these surface defects, including chemical etching, microlithography, or the relaxation of a very thin and highly lattice-mismatched film grown on the substrate that forms a periodic array of nanoscale islands. Further tailoring to achieve desired nanostructure orientation or composition is possible by applying an electrical or magnetic field during nanostructure growth.

Characterization of the intersection of these 1D nanostructures is expected to reveal new information about the structure, chemistry, and properties of this junction. Scanning electron microscopy and transmission electron microscopy are the primary tools that we plan to use in characterizing the tailored multidimensional nanostructures.

FY 2006 Accomplishments

ZnO was chosen as the material from which nanostructures were grown due to the experience and expertise the Georgia Tech collaborators had with it. We grew ZnO nanostructures from ZnO powder placed on a substrate in a tube furnace using chemical vapor deposition (CVD) and vapor phase evaporation (VPE) techniques. We grew the nanostructures by first heating the powder in a tube furnace to 800 °C for 20 minutes and then to 1400 °C for 60 minutes using Ar/N_2 as a carrier gas, a gas flow rate of 50 sccm, and a vacuum of 300 mbar. We then air cooled the tube furnace to room temperature.

Among the variables we studied in the growth of ZnO nanostructures were substrate patterning, surface roughness, and catalytic nucleation sites. We patterned silicon substrates either by NIL or focused ion beam (FIB). We discovered the following about the influence of substrate patterning, surface roughness, and catalytic nucleation sites on ZnO nanostructure growth:

- *Growth on NIL-patterned silicon substrates*: The pattern consisted of parallel lines of rectangular trenches (pitch 1 µm, trench width 0.4 µm, trench depth 0.5 µm). With no nucleation sites introduced onto the substrate, there appeared to be random deposition of ZnO nanostructures. However, we observed ZnO nanobelts bridging the trenches. There also appeared to be more growth of 1D ZnO nanobelts and nanowires in areas of the silicon substrate patterned with trenches than in areas without patterning. This suggests that the roughness or corrugation of the substrate surface can enhance ZnO nanostructure growth.
- *Growth on FIB-patterned silicon substrates:* The pattern consisted of parallel trenches (pitch 5 µm, trench width 4 µm). We examined five different trench depths: 1 µm, 500 nm, 250 nm, 100 nm, 25 nm. Few nanostructures grew in the patterns having deeper trenches (higher aspect ratio). FIB-patterned trenches had residual Ga on the trench surfaces, which appear to act as catalysts for promoting the growth of ZnO nanostructures.

Noting the role of catalysts in the growth of ZnO nanostructures, we selectively coated the patterned substrate surface with Au electrodes along the walls of the trenches using photolithography. We chose hydrothermal synthesis of the ZnO nanostructures for the Au-coated substrates, as the Au called for a low-temperature method of ZnO nanostructure synthesis. While slower than the CVD and VPE techniques, hydrothermal synthesis required a maximum temperature of only 90 °C.

The use of hydrothermal synthesis resulted in ZnO microrods having a relatively thick (~ 3 - 5 µm) diameter and hexagonal cross section. We grew the ZnO microrods on NIL-patterned substrates having either parallel lines of rectangular trenches (pitch 1µm, trench width 0.4 µm, trench depth 0.5 µm) or V-shaped trenches (pitch 1 µm, trench depth 0.5 µm). We discovered that the axis of the ZnO nanorods tend to grow normal to the plane of the Au coating. The use of a combination of patterned substrates with a catalyst seems to hold promise for the growth of tailored nanostructures.

Significance

The ability to custom design multidimensional nanostructures from different materials adds flexibility to the range of "tools in the toolbox" and opens up new possibilities in form, function, and flexibility to the range of nanoscale materials and structures that can be used to support national security missions. Some examples of applications of tailored multidimensional nanostructures that address Sandia's and DOE's national security missions include:

- Microfluidics – 2D and 3D nanotube structures can expand the range of geometries that can be used in microfluid routing systems that test and protect our water supplies under the homeland security mission.
- MEMS – The ability to create custom multi-dimensional nanostructures adds to the tools in the toolbox that can be used to build structures, nanomachines, and devices that support Sandia's nuclear weapons (NW) program.
- Waveguides – The missions of Sandia's Defense Systems and Assessments (DS&A), NW, and Homeland Security and Defense strategic management units may potentially benefit from the linear and planar waveguides that can be formed from the intersection of multiple 1D nanostructures or the intersection of nanosheets, respectively.
- Nanoelectronics – Multidimensional nano-structures can be used as interconnects in multilayered electronic devices developed for use in Sandia's NW and DS&A missions.
- Sensors – Small changes in heat, pressure, applied electromagnetic field, or other stimuli can cause a multidimensional nanostructure to expand, contract, or bend in a way that it acts as a nanoscale sensor or switch by forming a contact to complete a circuit. The nanoscale size of these tailored structures can increase the range of sensitivity in sensor applications that support homeland security programs.

Scanning Electron Microscope Doppler Vibrometer

93542
P. L. Reu, P. G. Kotula, B. D. Hansche

Project Purpose

As engineering challenges grow in the ever shrinking world of nanodesign, methods of making dynamic measurements of these materials and systems will become important. Electron microscopes have imaged these extremely small samples for years but are incapable of measuring dynamic events. We envision a means to measure these nanoscale dynamic events by converting an electron microscope into a Doppler velocimeter. This idea proceeds from the analogous concept of laser Doppler vibrometry. However, the obvious solution of using a laser to probe at the nano-scale is not feasible because the diffraction limit of light is orders of magnitude larger than the samples of interest.

The purpose of this feasibility project was to investigate the theoretical underpinnings of using electron beams for Doppler measurements. While success of the concept is not guaranteed, initial indications regarding the concept are positive. Possible practical pitfalls include electron source beam coherence and current available at the detector that may limit detection speeds and system bandwidth.

If answers to these problems can be found, the invention of the Doppler electron velocimeter (DEV) could yield a completely new measurement concept at atomistic scales. In addition to the very practical aspects of making dynamic measurements, a number of interesting quantum mechanics ideas regarding the interference of electrons, electron wave coherence, and fermionic behavior could also be investigated.

FY 2006 Accomplishments

The literature review we completed in the area of electron holography and Doppler electron measurements revealed that only one experiment has been conducted in Doppler electrons in the last 30 years. During that time period, electron holography was thoroughly investigated and successfully used but only for static measurements and typically with the intent of improving microscope resolution rather than making dynamic measurements.

Significance

We completed a SAND report on the theory of DEV and presented it as a late-breaking poster at the international conference on Microscopy and Microanalysis in Chicago, obtaining feedback from a number of practicing holographers. The report was positively reviewed within Sandia by electron microscopy and quantum mechanics experts, and an additional outside review by a published expert in electron holography was also positive, with some caveats regarding beam current and coherency; topics that were raised in the report. This work has positioned the idea for a full LDRD proposal in the next submission round.

The DEV is a completely new measurement concept. The possibility of making nanoscale dynamic measurements has not been attempted up to this point. Dynamic measurements in this context, using electron beams rather than lasers, also implies dynamic electric and magnetic fields may also be measured in addition to surface velocities. If successful, this new tool could have important impacts in the emerging nanosciences field. The ability to measure dynamic motion has been important in microscales and is expected to be equally important in the nanoregime. Beyond measurement of small devices, the possibility of measuring evolving material processes under different conditions also seems to be important.

To realize this concept, outside resources will be needed to supply the equipment and expertise in electron holography lacking at Sandia. We envision collaborations with Oak Ridge National Laboratory, a recognized leader in this field, as well as universities.

This new idea could provide a renaissance in electron holography and dynamic nanoscale devices. Beyond applied research goals, fundamental scientific investigations may also be pursued using the Doppler electron concept. These include quantum mechanical concepts of particle beam coherence and fermionic interference behavior. New insights into the double slit experiment may be elucidated regarding the "which way" types of arguments.

Other Communications

P.L. Reu, "Development of the Doppler Electron Velocimeter - Theory," presented at Microscopy and Microanalysis 2006, Chicago, IL, Aug 2006.

Si Nanocrystal as Device Prototype for Spintronics Applications

93543
W. Pan, M. S. Carroll, R. G. Dunn

Project Purpose

Silicon is the foundation of the modern information society. The whole silicon microelectronics industry is built upon the invention of the solid-state field-effect transistor (FET). However, the increasing miniaturization of microelectronics, the demands of optoelectronics, and the development of optical data transmission have shown the limits of silicon technology. New device physics is needed for next-generation information process and storage.

Recent years have seen the advent of spintronics with combined magnetism and solid-state electronics via spin-dependent transport process. Conventional electronics devices move electronic charge around. These novel spintronic devices manipulate both charge and spin degree freedoms by external means. Spintronic devices have the potential to operate at considerably higher speeds and consume less power than do conventional devices.

Before the spintronic device can become a viable technology, we have to answer the following questions: Can we combine ferromagnetic metals and semiconductors in integrated circuits? If yes, can we make magnetic semiconductor devices that work at room temperature?

Among the many approaches of combining magnetic ions and semiconductors, the dilute magnetic semiconductor of epitaxially grown III-V semiconductor, $Ga_{1-x}Mn_xAs$, has attracted the most attention. The Curie temperature, as high as 150 K, has been achieved in this material system. However, for real applications, any device has to be able to function at room temperature.

A critical hurdle to producing an operating room temperature spintronic device is to maintain the spin polarization and coherence. Recently, an above room temperature ferromagnetism in manganese doped silicon has been demonstrated. This remarkable result is extremely stimulating because it suggests that a critical component for spin-based FET may be achievable in silicon, making it better suited for spin-dependent transport process.

In this research project we examined the photoluminescence (PL) of Si-ncs (silicon nanocrystals) grown under different conditions and treated under different thermal budget. Experimental results from our Si-nc samples suggest that both the quantum confinement effect and the radiative interface states play an important role in PL.

We also studied magnetophotoluminescence and its temperature dependence in Mn doped si-ncs embedded in silicon oxide (SiO_2). This structure is selected because of the expected magnetic field dependence of the singlet-triplet state formed by the exchange interaction of electron and hole in Si-ncs, which should be controllable through external magnetic fields and internal magnetic fields produced, for example, by the Mn concentration in the material. This approach combines the advantages of PL measurements and quantum confinement effects and may, therefore, offer a unique ability to selectively pass certain spin polarizations in Mn doped Si-ncs.

FY 2006 Accomplishments

First, we successfully grew and characterized Si-ncs. Si-ncs are formed through a combination of high-density plasma enhanced chemical vapor deposition (HDP-CVD) of silicon rich oxides (SRO) followed by phase separation of the SRO into pure Si-nc and stoichiometric oxide during relatively long annealing (1-3 hours) at temperatures between 1000 °C to 1200 °C.

We characterized the Si-ncs and SROs using transmission electron microscopy, Fourier transmission infrared spectroscopy, Rutherford backscattering, nuclear reaction analysis, and ellipsometery. We studied the Si-nc growth in a relatively long thermal budget regime compared to most previous reports available in the literature. Morphology, defects within the nanocrystals, and size dependence as a function of thickness in the oxide are exaggerated in this regime and are more readily characterized in the longer diffusion length regime.

In particular, we observed evidence and implications of solid-phase epitaxial growth of the Si-ncs. We found that the effect of nearby silicon sinks at the surface and the substrate are highlighted by a strong nanocrystal size dependence on position in the oxide. The depletion of the oxides correlates with the nucleation of extended defects in the silicon substrate, which also could pose challenges to integration of the Si-ncs with other silicon devices.

In addition, because the Si-ncs depend on local geometries due to local silicon sinks, integration considerations become more complex when trying to target certain nanocrystal sizes in device structures. Finally, there are a number of studies of Si-nc growth using plasma enhanced CVD-produced SROs; however, few reports exist on the use of HDP-CVD SROs. This work, therefore, also offers additional experimental details related to the impact of using this different plasma geometry to generate the SROs.

Second, we carefully studied the PL in more than 20 Si-nc samples under different thermal budgets. We observed a very broad PL peak, blue-shifted from the bulk silicon band edge of about 1.1 eV. A surprising find was that in our samples, the PL peak is apparently pinned between ~ 900-1000 nm, independent of thermal budget. This is very different from previous studies, where the wavelength of PL peak can be tuned from ~ 600 to ~ 900 nm by controlling nanocrystal size. The pinning mechanism is currently unknown, though we speculate that it is probably related to the radiative interface states at the surface of Si-ncs.

In a further temperature dependence study carried out in a high thermal-budget sample, the PL intensity showed a nonmonotonic temperature dependence, similar to what has been previously reported. Postannealing in forming gas strengthens this nonmonotonic dependence, and the value of the temperature where the PL intensity displays a peak remains roughly the same, ~ 60 K. Based on the above observations, we believe that both the quantum confinement effect and the radiative interface states play an important role in PL.

Furthermore, we studied the magnetophotoluminescence in Mn-ion doped Si-ncs. We demonstrated, for the first time, evidence of possible ferromagnetism in Mn-ion implanted Si-ncs systems. This could lead to ultrafast, ultradense magnetic memory applications.

Significance

In light of future applications in integrated photonic circuits, highly luminescent nanostructured silicon materials, e.g., Si-nc, have attracted a lot of recent attention. In Si-nc materials, due to a strong quantum confinement effect, the momentum conservation rule is expected to be relaxed. Consequently, they can become efficient light-emitting materials.

Despite more than a decade of research, the physics of PL in Si-ncs remains unsettled. On the one hand, many experiments have shown that PL in Si-nc materials is modified by the quantum confinement effect. On the other hand, consistent with several previous experiments, our experiment showed that PL in Si-ncs is probably also related to the radiative interface states, e.g., the Si=O double bonds.

Another area with growing importance, and in which Si-ncs may play an important role, is using spintronics devices as sensors. Applications of these sensors are increasingly found in automotive, medical, and defense products and are expected to be important in homeland security applications. As an example, the ability to detect a small amount of magnetic field by using the spin-dependent tunneling process can find potential applications in detecting submarines.

Our demonstration of possible ferromagnetic order in Mn-ion doped Si-ncs may represent a significant step toward developing room temperature spintronic devices. It will unleash many more types of devices made possible by semiconductor high-quality optical properties and their ability to amplify both optical and electrical signals, for example, ultrafast switches and ultrasensitive sensors. Successful demonstration of spintronic devices is also of great interest for secure and fast telecommunication.

Spintronic devices also offer opportunities for investigating novel quantum transport effects. This effort is aligned with the nanoelectronics and photonics research of the Center for Integrated Nanotechnologies. By nurturing this science and harnessing it for spintronic technology systems, we can achieve a differentiating advantage for decades out.

Finally, this research will utilize the existing, world-class, leading-edge fabrication facilities at Sandia's Microelectronics Development Laboratory. It is relevant to an ongoing effort in silicon optoelectronics and is related to Sandia's solid-state lighting program and quantum information process research. Silicon is believed to be an ideal material system for solid-state computation because it is compatible with existing silicon fabrication technologies and because the zero nuclear spin and small spin-orbital interaction in bulk silicon help to reduce the spin-decoherence channels.

Refereed Communications

W. Pan, R.G. Dunn, M.S. Carroll, and Y.Q. Wang, "Experimental Studies of Photoluminescence in Mn-Ion Implanted Silicon Rich Oxide Thin Film," in *Proceedings of the Spring MRS Meeting*, pp. A08-08, April 2006.

Other Communications

M.S. Carroll, W. Pan, L. Brewer, J. Verley, J. Banks, R. Dunn, and T. Headley, "Squeezed Silicon Nanocrystals in SiO_x/Si_3N_4 Superlattices Grown with High-Density Plasma Chemical Vapor Deposition," presented at the American Vacuum Society Meeting (NM chapter), Albuquerque, NM, October 2005.

Electrochemical Sensing through Parallel Chemometric Diagnostics

93544
W. A. Steen, C. L. Stork

Project Purpose

Powerful data mining techniques such as multivariate curve resolution and data patterning, also known as chemometrics, have been developed largely to deconvolute spectral data. Applying these techniques to electrochemical data has only begun to be considered. Combining a suite of electrochemical techniques, chemometric protocols, and electrode arrays could create diagnostic tools for detection and analysis that are more powerful than state-of-the-art electrochemical sensors.

Current electrochemical sensors typically rely on specific interactions between an analyte and a small number of electrodes. The techniques used to probe the interactions are typically straightforward potentiometric, amperometric, or conductometric techniques. When techniques that offer more fidelity are used, they are typically used on a very small scale. Sensors that have a large number of electrodes, i.e., electrode arrays, usually rely on a cumulative signal rather than on individual responses. When elements of the electrode array are individually addressable, they are sequentially tested through multiplexing or relay switching schemes, which slows sampling time (i.e., parallel testing is very rare and used on a small scale).

The purpose of this project is to combine and build upon these isolated efforts to establish the foundation for a new generation of electrochemical sensors. A large number of individually addressable electrodes with tailored and engineered surfaces will be characterized, in parallel, using cyclic voltammetry. Data generated will then be analyzed with multivariate analyses techniques. Through this approach, composite interactions between many members of the electrode array and many analytes that have no otherwise observable trends can be quantitatively assigned to a particular analyte using chemometric protocols. Furthermore, the interactions need not be well understood, provided they are mathematically significant.

FY 2006 Accomplishments

We successfully demonstrated the usefulness of applying multivariate techniques to electrochemical data by creating an electrode array and interrogating monosaccharide solutions. This model system can ultimately guide creation of next-generation electrochemical sensors. Specific accomplishments include:

- Decorating a 10 x 10 array of platinum electrodes with 10 different electrodeposited platinum-ruthenium alloys
- Interrogating solutions of pure glucose, galactose, and fructose over the array and applying principal component analysis (PCA) to the resulting data sets. PCA was able to successfully classify each sugar based on cyclic voltammetric data.
- Created partial least-squares regression models for predicting glucose, galactose, and fructose concentration from 15 ‘training’ solutions. Each training solution contained all three monosaccharides in concentrations ranging from 10 - 1000 mM. The model was based on cyclic voltammetric data.
- Used partial least squares (PLS) models to predict monosaccharide concentration of 12 ‘test’ solutions. These solutions also contained the three monosaccharides. The values for the root-mean-squared-error-of-prediction (RMSEP), a quantitative measure of the predictive accuracy of a PLS model, between 10 - 1000 mM were 142 and 120 mM for the glucose and galactose models, respectively. The RMSEP for the fructose model was 128 mM from 10 - 500 mM. These values, approximately 15 percent of the maximum concentration, are very encouraging for having so few ‘training’ solutions (15) and such a large concentration window (10 - 1000 mM).

We documented these results in an article we recently submitted to *Journal of the Electrochemical Society.*

Significance

The accomplishments of this project and follow-on work can help explore electrochemical material science issues related to virtually every laboratory mission area. Homeland security is the most obvious benefactor from advanced electrochemical sensing, as monitoring and protecting our nation's water supply is critically important. Other applications of electrochemical sensors include industrial process streams (both government and private sector), corrosion monitoring (which impacts national defense via stockpile stewardship), and medical applications (glucose sensing). The science and technology community, as it relates to the electrochemical sciences, benefits through the further application of multivariate techniques to electrochemical data.

The results of this project will be leveraged in a new LDRD project beginning in FY 2007 to further investigate and exploit multivariate electrochemistry.

Biological Detection and Tagging Using Tailorable, Reactive, Highly Fluorescent Chemosensors

93545

J. R. McElhanon, T. Zifer, L. A. Rahn

Project Purpose

The purpose of this project was to create a versatile chemosensor family for rapid, sensitive, and unambiguous labeling and detection of biological and chemical target molecules. Long-wavelength fluorogenic chemosensors that are reactively activated by biological agents would provide new opportunities in sensitivity and selectivity for bioscience applications. Other important fluorescent markers (e.g., green fluorescent protein and derivatives) in cell biology are costly and suffer from background fluorescence from unreacted probes in experiments designed to detect molecular interactions.

Our objectives were focused on the development of a fluorogenic chemosensor family that could be tuned for reaction with electrophilic (e.g., chemical species, toxins) and nucleophilic (e.g., proteins and other biological molecules) species. Our chemosensor approach used the fluorescent properties of well-known berberine-type alkaloids. In situ chemosensor reaction with a target species transformed two out-of-plane, weakly conjugated, short-wavelength chromophores into one rigid, planar, conjugated, chromophore with strong, long-wavelength fluorescence (530-560 nm) and huge Stokes shift (100-180 nm). This approach was amenable to tuning fluorescence intensity, reactivity, wavelength, and Stokes shift through altering the substituents on the chemosensor molecule. The real challenge of this project was determining biospecies specificity.

FY 2006 Accomplishments

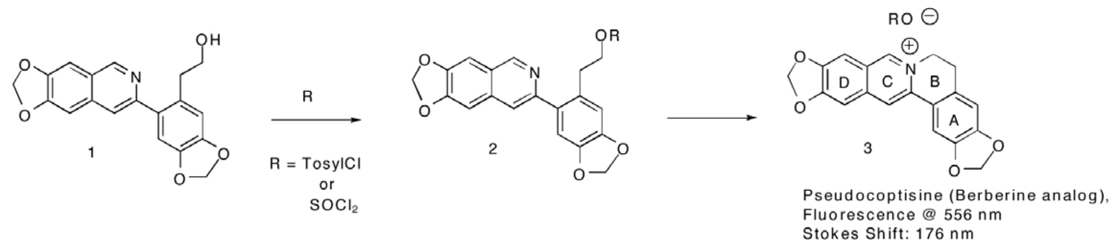
Our tagging and detection approach involved preparing appropriately functionalized precursors that were reactive toward nucleophilic biomolecules. We demonstrated proof-of-principle experiments (Scheme 1) for reaction of fluorogenic chemosensor **1** with two electrophilic species, *p*-toluenesulfonyl chloride and thionyl chloride. Reaction of **1** with either species resulted in formation of pseudocoptisine **3**, a highly fluorescent berberine analog.

Pathways to the preparation of **1** and a family of related fluorogenic sensor molecules were well established in our laboratories. Alcohol **1** contained two out-of-plane, weakly conjugated, short-wavelength chromophores. Once **1** reacted with a target species, intermediate **2** formed, followed by an intramolecular cyclization reaction resulting in a rigid, planar, conjugated, highly delocalized chromophore, pseudocoptisine **3**.

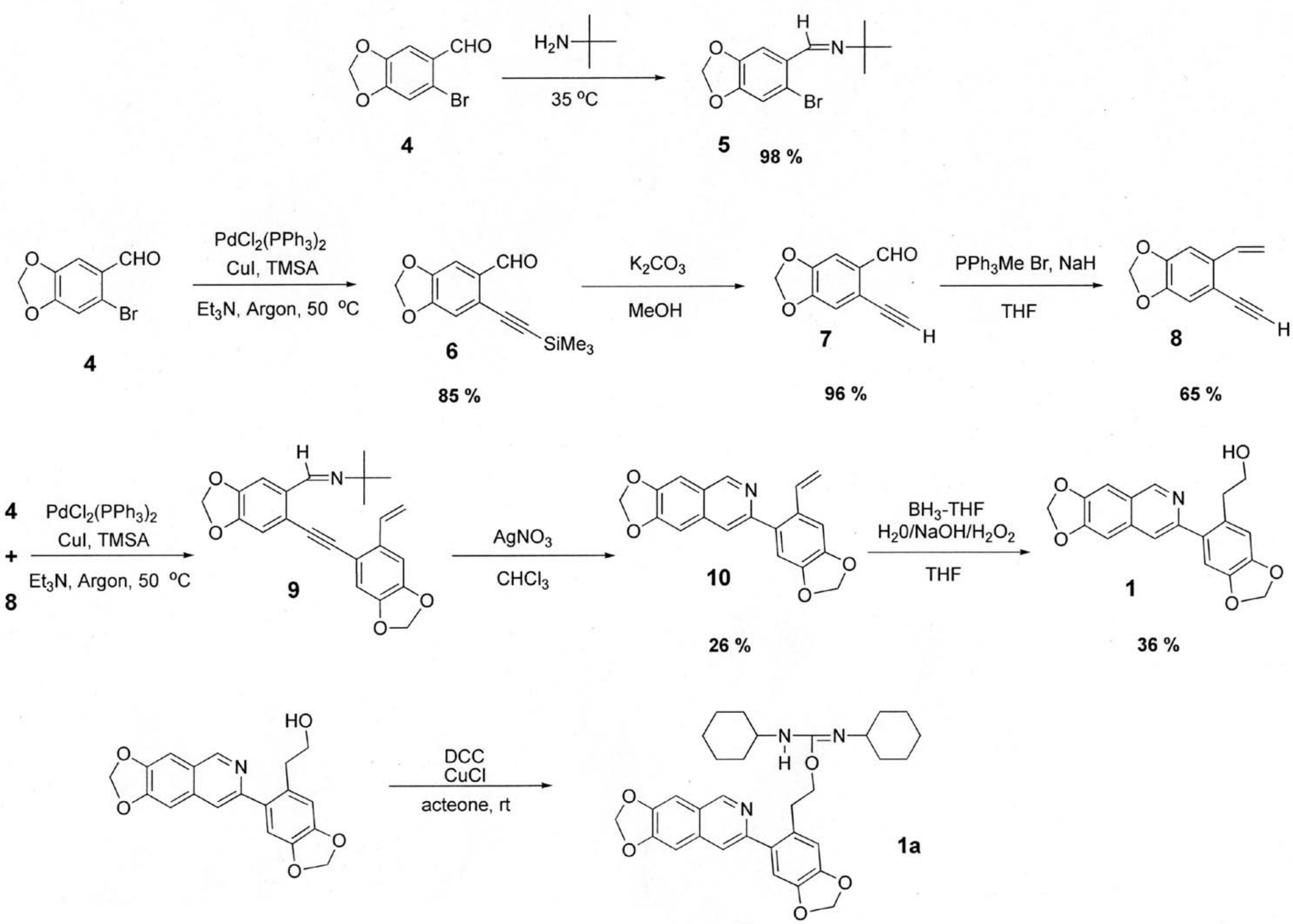
For nucleophilic reaction and detection of biomolecules, **1** required activation of the alcohol functionality for reaction with such molecules. Carbonyldiimidizole (CDI) is a known linker for cross-linking biological molecules (proteins, antibodies, and DNA) and modifying polymeric substrates such as poly(vinyl alcohol) for chromatographic applications [1]. Other reports had suggested that the carbonyl group of carbamates similar to **1a** (Scheme 2) are unreactive toward nucleophilic attack by the nitrogen of the quinoline group, therefore ring closing of **1a** to form **3** was not anticipated. **1** was reacted with CDI to generate carbamate **1a** and was identified by thin layer chromatography. Unfortunately, **1** was a transient species and underwent the nucleophilic ring closing cyclization to pseudocoptisine **3** as observed by the formation of a fluorescent yellow spot at the origin of the thin layer chromatography plate.

Dicyclohexylcarbodiimide (DCC) can also be used for activation of **1** toward biomolecules and was pursued in parallel with CDI. Reaction of **1** with excess DCC and a catalytic amount of CuCl in acetone resulted in complete conversion to isourea **1a** as observed by ^{1}H nuclear magnetic resonance (NMR). **1a** was found to be unstable in acetone solution when left for several days or when concentrated in vacuo and stored. Conversion to ring-closed product **3** and degradation occurred when stored under these conditions. However, stable solutions of **1a** could be prepared through in vacuo concentration of the reaction mixture followed by immediate redissolving in petroleum ether.

Isourea **1a** was reacted with the amino acids aspartic acid and lysine in deuterated dimethylsulfoxide (DMSO-d6) solution and monitored by NMR. Complete conversion to pseudocoptisine **3** occurred in approximately six hours. Treatment of **1a** with lysine also required longer reactions times, approximately 48 hours for complete conversion to pseudocoptisine **3**. The longer reaction times of the amino acids were attributed to their poor solubility in DMSO, unlike benzoic acid, which was readily soluble. We conducted ^{1}H NMR control studies with aspartic acid and lysine in DMSO-d6, verifying the insolubility



Scheme 1. Electrophilic proof-of-principle reaction yielding highly fluorescent berberine analog



Scheme 2. Synthesis of Nucleophilic Chemosensor **1a.**

of the amino acids under these experimental conditions. It is likely that mixtures of DMSO and water would improve solubility and reaction rate of the amino acids. Absorption and fluorescence spectra were obtained from reaction of **1a** with aspartic acid and lysine and are consistent with formation of pseudocoptisine **3**.

Significance

This work has demonstrated the design, synthesis, and function of a reactive chemosensor for the detection of biomolecules based on the formation of fluorescent berberine-based natural products. The chemosensor described has been tailored with a reactive isourea coupling group that is reactive with molecules containing carboxylic acid residues. Reaction of this fluorogenic sensor with benzoic acid resulted in a rapid (< 3 minute) detection response time as observed by ^{1}H NMR. Amino acids aspartic acid and lysine were also detected by ^{1}H NMR and fluorescence spectroscopy; however, detection times were slower due to the solvent conditions employed. Further work is needed to elucidate the fluorescence response of the chemosensor.

With further study, this work would likely lead to a rapid, sensitive detection and discrimination method for the detection of a variety of chemical species, proteins, and other biomolecules.

[1] S. Pathak, A.K. Singh, J.R. McElhanon, P.M. Dentinger, "Dendrimer-Activated Surfaces for High Density and High Activity Protein Chip Applications," *Langmuir*, vol. 20(15), pp. 6075-6079, July 2004.

Self-Cleaning Synthetic Adhesive Surfaces Mimicking Tokay Geckos

93547
C. J. Brinker, E. D. Branson, S. Singh, J. E. Houston

Project Purpose

Close examination of gecko feet reveals a remarkable hierarchy of structures. At the coarsest (millimeter) scale we see lamellar-like features oriented perpendicular to the direction of motion. The lamellae are composed of arrays of setae, rod-like structures (100 µm long x 4 µm in diameter) that each branch into ~ 1000 finer fibers (10 µm long 0.1 µm wide), terminating in leaf-like plates called spatula.

This design presumably makes the lizard adhesive system elastically compliant/adaptive over the range of length scales needed for locomotion/adhesion on surfaces with varying roughness and surface chemistry. This hierarchical design also allows for rapid deadhesion that occurs by a successive peeling process. The purpose of the project is to design a new class of synthetic adhesive surfaces of potential interest for conferring Gecko-like functionality to man-made devices.

A major activity of this project was to devise processing strategies to develop hierarchical surfaces with systematically varying structural features.
A second major activity was to measure adhesive behavior and to develop relationships between adhesion and structure. Interfacial force microscopy (IFM) was used to generate force-displacement profiles. By performing measurements using materials fabricated with a wide range of fiber feature sizes, we attempted to establish the relationship between feature size and pull-off force for this new class of synthetic materials.

FY 2006 Accomplishments

We used two lithographic approaches to make hierarchal structures with dimensions similar to the gecko foot dimensions.

One approach combined photolithography (PL) with soft lithography (micromolding) to create surfaces with systematically varying dimensions, spacings, and areal densities of both rod-like and fiber-like features. The fabrication process used anodic alumina membranes as micromolds to define the smaller-scale features of the fibers and PL to define the coarser-scale features corresponding to the rods.

We deposited SU-8, an EPON™ epoxy-based resin, on a substrate and placed an anodic alumina membrane on top of the SU-8 that would allow the SU-8 to wick up through the membrane. Ultraviolet (365 nm) irradiation through a mask positioned on top of the SU-8 overlayer defined both the rods and the fibers. Standard baking and solvent steps cured the SU-8 and removed unexposed regions (defining the rods); dissolution of the alumina micromold using hydrochloric acid defined the fibers. In this fabrication scheme, the fiber feature size, defined by the alumina micromold, was 0.2 µm in diameter x 60 µm height periodically spaced in a square lattice with spacing on 0.75 mm squares 5-10 microns thick.

The second approach followed more conventional PL-based patterning. Initial experiments used interferometric lithography to pattern a photoresist coated glass substrate. We performed standard metal lift off, resulting in a chrome-coated mask with arrays of ~ 0.3 µm holes on a ~ 1.0 µm spatial period. We used these masks in two separate schemes: 1) SU-8 resist was spun directly on the glass substrate/mask and exposed from the backside such that the resist over the hole was exposed and remained upon postexposure bake and develop, and 2) the mask was used as a typical contact printing mask to pattern other samples with photoresist. In both cases, patterned features with dimensions ~ 0.3 mm in diameter by 0.5 mm tall were produced. The feature height is a function of the resist thickness upon spin, while the diameter is controlled by the mask dimensions.

Based on this initial success, we will continue to pursue higher aspect ratio structures by further optimization of the resist processing and exposure parameters.

We used IFM to measure surface adhesion on both advancing and retracting surfaces, using a diamond tip with a diameter of 200 nm. The measured adhesive forces ranged from 2.65 to 5.33 $\mu N/\mu m^2$.

Significance

We developed characterization tools, processing skills, and fabrication techniques that led to the development of novel nanostructures that may be useful for applications requiring reversible adhesion and high strength to surface area ratios. We fabricated a hierarchical structure using a combination of lithography and micromolding to demonstrate novel structures that may satisfy these requirements. The IFM was a very effective tool for initial quantitative measurements of surface adhesion.

Other Communications

P. Johnson, S. Singh, E. Branson, J. Houston, and C.J. Brinker, "Synthetic Adhesive Surfaces Mimicking Geckos," presented at the Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2006.

Nanoengineered Electroluminescent Polymers

93548
C. H. Fujimoto

Project Purpose

Existing light sources, such as incandescent lamps and bulbs, are extremely inefficient in that only 30 percent of the energy consumed is used to generate light; the rest is wasted as heat. In order to increase efficiencies, new lighting technologies are needed. Organic light emitting diodes (OLEDs) and polymer organic light emitting diodes (p-OLEDs) show promise in replacing incandescent sources, as OLEDs have been projected to be twice as efficient as conventional light sources. Experimental OLEDs are already more energy-efficient than incandescent lamps.

In 2002, the DOE Office of Building Technology and the Optoelectronic Industry Development Association (OIDA), through public-private partnerships, published a technology roadmap for developing OLED/p-OLEDs. This roadmap indicated that many challenges still must be overcome in both OLED and p-OLED before commercialization. Some of these critical issues were the development of highly efficient luminescent materials; the development of stable, highly conductive electrodes; and the development of OLEDs with high internal quantum efficiencies.

The advantages of p-OLEDs over OLEDs include lower processing costs and the ability to be fabricated into large surface area displays, which may be difficult with small molecule OLED technologies. A disadvantage of p-OLEDs is the lack of stable, highly conductive blue luminescent polymers. This project addresses this hurdle to developing new, high-charge transport p-OLEDs.

FY 2006 Accomplishments

- Successful synthesis and characterization of four unique poly(phenylene)s through Diels-Alder polymerization
- Successful fabrication of dual-layered p-OLEDs with newly synthesized polymers
- Increased understanding of the light emitting properties of synthesized polymers and their structure-property effects.

Significance

Although a proven family of polymers, Diels-Alder poly(phenylene)s have not been used for p-OLEDs and represent a potential "cutting edge" material for this application. The results of this project are extremely promising and show that this class of polymers emits blue light when a voltage is applied. The discovery of a new class of blue light emitting polymers will impact Sandia's goal to prepare for a crisis in global energy supply.

Engineering Intracellular Active Transport Systems as In Vivo Biomolecular Tools

93549
G. D. Bachand, A. Carroll-Portillo, M. Bachand, A. C. Greene

Project Purpose

Active transport systems provide essential functions in terms of cell physiology and metastasis. These systems, however, are also "hijacked" by invading viruses, which enables the virus to be transported to and from the cell's nucleus (i.e., the site of virus replication). Such directed trafficking of macromolecules by active transport systems alleviates the diffusional constraints associated with the highly viscous environment found in the cell's cytoplasm. While these systems are essential for cell metastasis, new approaches for interrogating the inner workings of living cells may be achievable by "co-opting" the active transport system in a manner analogous to those common with viruses and other microorganisms.

The cytoskeleton of eukaryotic cells consists of a three-dimensional (3D) network in which motor proteins can actively move throughout the volume of a cell. One specific type of cytoskeletal filament, called microtubules, organizes around a common center, called a centrosome, and forms a polar network filling the 3D volume of a cell. Two distinct classes of motor proteins, dynein and kinesin, use microtubule networks to achieve efficient, bidirectional transport of macromolecules within the cell.

The overall goal of this project was to explore the ability to use kinesin and microtubules as a means for selectively binding and transporting target biological molecules within the confines of a living cell. This technology may offer the ability to harvest rare intracellular targets in situ, providing significant advantages over standard methods that often dilute rare analytes to concentrations that cannot be determined by current methods. Quantifying changes of such rare analytes may in turn further our understanding of cell physiology, including the details of innate immunity.

In addition, the proposed system potentially could enable selective disruption of the transport systems, and consequently the infection and pathogenesis cycle of virus, or enable targeted delivery of therapeutic agents.

The scientific challenges involved in achieving the overall goal of this project included (1) engineering kinesin motor proteins capable of selectively binding target molecules and (2) applying these engineered kinesin constructs to live eukaryotic cells. Thus, the specific objectives of this project were to develop a means of integrating selective elements with kinesin and to demonstrate in vitro capture of target molecules using this active capture and transport system.

We engineered a chimeric fusion protein consisting of kinesin (motor protein) and a single chain variable antibody fragment (selective element) and evaluated the functionality of these constructs. We then developed and characterized several methods of harvesting the intact microtubule networks from eukaryotic cells.

FY 2006 Accomplishments

The first objective of this work was to construct a chimeric fusion protein consisting of the *Drosophila* kinesin motor protein and a single chain variable fragment (scFv) of an antibody.

We obtained a plasmid containing an scFv specific against rabbit immunoglobulin G (IgG). We used site-directed mutagenesis (SDM) to genetically modify the scFv sequence to introduce a NarI restriction endonuclease site to facilitate the insertion of the scFv fragment into the kinesin expression system.

We excised the modified scFv sequence and *Drosophila* kinesin sequence (pPK113-DM) by digestion with NarI and NotI. The scFv and Drosophila kinesin fragments were ligated together, and transformed into XL1-blue cells. The resulting kinesin-scFv construct contained a frameshift that was rectified by deleting a thymidine residue at the 5' end of the scFv.

Finally, we introduced a 10x His tag and hard stop codon (TAA) to provide an easy means for protein purification and proper protein length, respectively. DNA (deoxyribonucleic acid) sequencing of the final construct was performed by Northwoods DNA, Inc. We analyzed the sequencing results to verify proper placement and orientation of the individual regions.

The kinesin-scFv was expressed in BL21 pLysS cells and purified by affinity chromatography. The purified protein had an estimated molecular weight of about 140 kDa, consistent with the predicted molecular weight based on the sequence.

We evaluated the avidity and antigenic selectivity of the kinesin-scFv using enzyme-linked immunosorbent assay. We effectively bound rabbit IgG to the kinesin-scFv and detected it at levels as low as 1 ng/mL, confirming the preservation of antibody functionality. Using a double enzyme spectrophotometric assay, we observed the decreased level of ATP (adenosine triphosphate) hydrolytic activity of the kinesin-scFv, compared to the wild-type kinesin. Together, these data demonstrate the successful construct of a chimeric kinesin motor protein capable of selectively binding target analytes.

We successfully prepared intact microtubule networks from both the human liver (HeLa) and rat neuronal (B35) cells using cross-linking (glutaraldehyde) and fixation (methanol) methods. Epifluorescence microscopy of these networks demonstrated the removal of the cell membrane, as well as all intracellular organelles (e.g., mitochondria, nucleus, and so on) and the actin cytoskeleton.

This methodology represents a key enabling technology for this project as well as a range of nanotechnological applications. For example, these microtubule networks can be used to study the kinetic rates of transport and analyte capture, on which models can be developed to predict functionality in live cells. In addition, the cell's microtubule architecture can be applied to hybrid nanoscale systems as a 3D transport network that may be used to synthesize structured materials.

We did not achieve the implementation of microtubule networks in bead-based, kinesin motility assays during the course of this project. Attachment of kinesin-coated fluorescent microspheres and quantum dots to microtubules was observed by fluorescence microscopy; movement, however, was not demonstrated for microtubule networks prepared by either cross-linking or fixation. A number of factors could have potentially limited kinesin function, including the presence of microtubule-associated proteins and excessive cross-linking of tubulin molecules.

Significance

The accomplishments resulting from this project may be leveraged by existing programs at Sandia, as well as by new, emerging projects. The kinesin-scFv construct developed in this project represents a new tool that can be used to study host-pathogen interactions for national security applications. More specifically, our accomplishments offer the ability to engineer a "library" of analyte-specific motor proteins that may be used to study the inner workings of a cell.

The target application of this technology is the integration of kinesin-scFv constructs with immune cells as a means of capturing rare signaling molecules in situ. Initial stages of the innate immune responses involve a range of small molecules (e.g., transcription regulators) that experience only a slight shift in concentration or state. For example, nuclear transcription factor-kB (NF-kB) exists in at least 15 discrete forms, each of which plays a role in regulating gene transcription during the early stages of an immune response. The relatively small change in form of NF-kB during signal transduction has considerably limited our ability to resolve specific roles for the different forms.

A significant hurdle limiting our understanding includes the inability to synchronize the immune response among cells within a population, which in turn leads to noisy, imprecise data sets. Thus, in order to fully understand pathogen-induced immunological response, we must interrogate changes in proteins and small molecules within individual cells. However, the ability to detect such rare analytes at the single cell level, particularly in the context of a large, heterogeneous background, is not possible with current technology.

Application of the kinesin-scFv construct for the in situ harvesting of target analytes offers a number of key advantages. For example, kinesin-based active transport within the cell removes diffusional constraints associated with the gel-like cytoplasm. Kinesin transport also enables a systemic means of probing the 3D volume of a cell, moving radially outward from the center.

The technology developed in this project forms the basis for a new LDRD project focused on specifically applying kinesin-based harvesters to study changes in NF-kB levels within a cell. Further development of this technology may benefit efforts such as the Microscale Immune Study Laboratory Grand Challenge LDRD project, and provide a basic understanding of host-pathogen interactions.

The ability to prepare intact microtubule networks from living cells represents a key enabling technology with a broad a range of applications. This methodology will be used in the new LDRD project as a means for studying the kinetic rates of transport and analyte capture. These microtubule architectures can be applied as a 3D transport network for synthe-sizing dynamic, nanostructured materials.

This technology could be useful in an existing project sponsored by DOE's Office of Basic Energy Science, which focuses on understanding and developing dynamic nanocomposite materials based on kinesin transport systems. The 3D microtubule network is a fundamental scaffold that can be used to mimic functions such as the color changing system found in fish and other organisms.

Self-Assembled Nanoexplosives

93551
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Project Purpose

The fabrication of traditional explosives typically starts from powders of energetic compounds that are tightly squeezed into random close-packings at high densities in a polymer resin. The higher the density, the larger the explosive power density. However, high-volume fractions come at a price, as the composite is more likely to explode prematurely and unintentionally. This degree of unpredictability is in part due to the randomness of the packing; it only takes a small cluster of particles to be touching or almost touching to create a so-called hot spot.

As we move toward smaller explosives, the problems associated with simply extending traditional assembly methods to the nanoscale become significant. For instance, nanopowders are extremely difficult to handle and control, and packing them tightly, or the challenge of mixing them with polymer resins, is likely to result in a much larger variability of the final product.

Our idea constitutes a radical deviation from the tried approach and revolves around taking advantage of the small scale by implementing versatile self-assembly (SA) techniques to organize the explosive material in three-dimensional (3D) space. We proposed to switch to a fabrication route that could ultimately be entirely solution based and, therefore, controllable by a host of solution-based strategies. We seek to both synthesize and assemble the energetic particles in a liquid environment. This enables us to take full advantage of all the solution SA tricks that have, in the past decade, allowed the creation of robust 3D organized films and mesophase particles.

FY 2006 Accomplishments

We used physical vapor decomposition by thermal evaporation through shadow masks to create 3D structures of three types of explosives (pentaerythritol tetranitrate [PETN], 1,3,5-trinitro-1,3,5-triazine [RDX], and keto-RDX [K-6]). Using crossed shadow masks, we created square holes of 300 micron and deposited PETN, K-6, and RDX explosives. RDX deposited slightly better than PETN, producing better defined features. Using square-hole masks with sizes varying from 200 to 600 microns, we performed additional depositions with PETN, K-6, and RDX.

The deposition results were strongly dependent on the explosive used. We observed extensive deposition underneath the mask when using K-6, but not with RDX. This may indicate a pronounced dependence on the wetting characteristics of the explosive in contact with the mask. RDX produced the best square shapes, with sharp features, showing distinct promise for the production of 3D energetic materials.

We investigated self-assembly with the intended explosives. We synthesized ordered-composite Au-nanocrystal/silica particles through self-assembly of nanocrystal micelles and silicate. Depending on the use of surfactants and the kinetic conditions of the silica hydrolysis and condensation, well-shaped or irregularly shaped particles formed. Inside of these, SA nanocrystals adopted a face-centered cubic mesostructure.

We moved the supercritical fluid expansion instrument to a new facility and reassessed the associated operating procedures and safety documentation. We identified a particulate inhalation hazard, and updated and revised the safety documentation, operating procedures, and engineering controls. These were completed and approved too late for use in preparing samples in FY 2006.

We used computational methods to study the packing behavior of bulk systems of cubes as well as spheres inside narrow cylinders, mimicking the behavior of deposition and drying of a nanoparticle solution inside a narrow channel. We observed a surprisingly rich phase behavior for these systems, with intricate well-defined spiraling structures developing as the diameter of the cylindrical channel varied from about 1 to 3 sphere diameters. The type of structures strongly influenced the local contacts between the individual grains. We performed similar calculations with the CTH code for short cylindrical particles packed in larger cylinders and detonated.

Significance

We were able to obtain pilot results that were useful for furthering the concept of using nanoscale self-assembly techniques for producing highly-packed explosives with improved explosive power and stability against premature and unintentional explosions.

We demonstrated that physical vapor decomposition by thermal evaporation through shadow-masks can be used successfully to create ordered 3D structures (with sizes ranging from 200 microns to 600 microns) of three relevant explosive materials: PETN, K-6, and RDX. This pilot fabrication effort is a useful step toward realizing test systems suitable for facilitating synergy between computational and experimental efforts and toward fabricating highly-packed 3D explosive nanostructures with improved power and explosive control/stability.

The demonstration of the fabrication of gold-silica self-assembled composites via inverse micelle fabrication was a useful step toward realizing the feasibility of alternate solution-based fabrication techniques for self-assembled nanoexplosives.

Finally, in collaboration with the Aberdeen Proving Ground, we used computational techniques to advance the study of the packing behavior of bulk systems of explosive cubes in narrow cylinders useful for advancing computational approaches toward nano-structured explosive materials.

A surprisingly rich phase behavior was predicted by these simulations, lending useful insight on controlling the hot spot issue vital for improving explosive stability and handling.

In summary, this project illustrated the potential viability for developing densely packed nanoexplosive materials in polymer resin materials (with acceptable handling and control properties) via novel solution-based techniques with guidance from modeling techniques. Researchers at military laboratories (like Aberdeen Proving Grounds) are interested in this nanostructured explosive materials approach, and the results have the potential to guide more extensive future efforts that seek improvement in nuclear weapon firing systems relevant for NNSA mission needs.

Refereed Communications

R. Banton, J. Starkenberg, and F. van Swol, "Simulation of Detonation Propagation and Failure in Granular Explosive Charges," in *Proceedings of the 13th JANNAF International Detonation Symposium*, July 2006.

H. Fan, J. Gabaldon, C.J. Brinker, and Y.B. Jiang, "Ordered Nanocrystal/Silica Particles Self-Assembled from Nanocrystal Micelles and Silicate," *Chemical Communications*, vol. 22, pp. 2323-2325, June 2006.

Nanoporous Silica Templated Heteroepitaxy

99405
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Project Purpose

The purpose of this project is to explore the use of porous growth masks as a method for defect reduction during heteroepitaxial crystal growth. High-quality heteroepitaxy, or crystal growth of one material system on a substrate of another material system, is a long sought goal that will continue to increase in importance.

GaN and AlN, important wide bandgap semiconductors as ultraviolet (UV) light sources and radio frequency (RF) electronics materials, are typically grown on sapphire or SiC due to the lack of suitable bulk substrates.

In addition, there is considerable interest in growing optoelectronic material systems on silicon substrates to incorporate and leverage mature silicon electronics processing with optical materials. The inevitable threading defects that result from the lattice mismatch in growing heteroepitaxially degrade both the optical and electrical performance of semiconductors.

Current research shows that growth on patterned substrates can significantly reduce defect density. Cantilever epitaxy and nanoheteroepitaxy (NHE), techniques with features on the micrometer and 100s of nanometer scales, demonstrated > 100 X reduction in defect density. The NHE theory predicts that features in the 10-20 nm range could offer defect-free heteroepitaxy in many important growth systems; however, patterning a substrate over large areas at these dimensions is not possible with current lithography techniques.

We planned to focus on using nanoporous surfactant templated silica as a growth mask to achieve the benefits of nanoheteroepitaxy. While we did some preliminary work on this material system, the scope of the research was expanded to a broader range of growth masks including mesoporous carbon and the UV-curable photoepoxy SU-8.

In contrast to conventional approaches to nanoheteroepitaxy – where an amorphous growth mask is deposited as a planar film, patterned using one of the available lithography approaches, and transferred to the substrate by plasma etching of the growth mask – all of the approaches we explored are single-step patterning solutions. As such, these “soft patterning” approaches require no energetic plasma etching of the amorphous layer, and thus avoid potential damage to the underlying single crystal substrate.

FY 2006 Accomplishments

First attempt to use a mesoporous carbon as a growth template

We spun a carbohydrate/block copolymer nanocomposite film onto a sapphire substrate. After pyrolysis in an inert environment, a mesoporous carbon film resulted. We placed this sapphire substrate in the growth reactor under standard GaN growth conditions. The result was a polycrystalline film, indicating that further work on this growth template is needed.

First attempt to use SU-8 as a growth template

SU-8 can be stable at temperatures up to about 700 ºC, above the nucleation temperature for GaN growth. Because of this, we attempted to use SU-8, patterned with interferometric lithography, as a direct growth mask. Using a one-step patterning approach avoids pattern degradation upon pattern transfer to the substrate as well as crystal damage to the underlying substrate from reactive ion etching typical in most hard mask processes for heteroepitaxy. The preliminary results indicate that faceted GaN, possibly single crystal, was grown, but that in the growth environment, the SU-8 pattern may be breaking down. We identified several different approaches that may solve this problem.

Pilot work on using SU-8 as a deep ultraviolet (DUV) negative photoresist

We performed some very early work on using SU-8, an i-line photocurable epoxy, as a DUV resist.

For films thinner than the absorption depth of SU-8, the resist can be patterned by DUV (193 nm) light. Patterning was successful; however, the patterns appear to suffer from adhesion issues, possibly related to stress in the deposited SU-8. We are continuing to investigate this very promising direction.

Significance

From a crystal growth standpoint, a successful single patterning step nanoheteroepitaxy approach such as those pursued in this research would provide an inexpensive method for producing higher-quality GaN as well as the possibility of incorporating III-V materials on a silicon substrate. As they are the starting materials for any GaN or III-V based application, the production of low-defect density substrates would have tremendous impact in a diverse range of fields including solid-state lighting, next-generation RF devices, spectroscopy sources, and highly integrated III-V optical materials on monolithic silicon electronics platforms.

As an ancillary breakthrough, the ability to use SU-8 as a negative DUV resist could be a valuable addition to research and industrial patterning applications. Current DUV resists are usually positive and, almost without exception, very sensitive to atmospheric degradation by airborne amines. SU-8 is a very durable resist both during and after application and requires no special handling.

The ability to pattern SU-8 with DUV sources, and hence generate patterns with feature sizes < 100 nm, opens up a wide range of science in a size scale falling between that of bottom-up approaches and most typical top-down lithography approaches.

Chiral Multichromic Single Crystals for Optical Devices

99406

R. A. Kemp

Project Purpose

We discovered an unprecedented class of chiral single crystals that are multichromic. We can prepare individual single crystals that are crystallographically identical, yet may be grown colorless, yellow, or green from crystal to crystal, or (and to our knowledge, for the first time) multicolored within the same crystal. These novel materials are based on s- and p-block elements (main group elements such as Ga and Al) that contain chiral chelating ligands.

Our prior work showed that chemical variations in the backbone ligands enable single crystals that maintain multichromism at all viewing angles and at reasonable temperatures; the materials are not doped but appear subtly altered structurally during growth. The purpose of this project is to investigate the mechanism for these color changes, in particular multicolored facets that occur within the same crystal, so that we can control and exploit these phenomena for optical materials applications.

Compounds with chiral ligands are of interest for several reasons, among them uses as nonlinear optical materials and as chirality-sensitive templates for biological sensors. The current materials appear polar and show promise for nonlinear optical (laser frequency doubling) materials with a tailorable polarization. Additionally, the ability to alter the chemical backbone of crystals should enable targeted environmental sensors, whereby the periodicity of the chiral structure may be tailored to match analytes of interest through choice of chelating ligands. It is rare that an entirely novel class of materials is invented that so well matches Sandia's interests in materials and optical applications.

FY 2006 Accomplishments

We synthesized and crystallographically characterized a number of these compounds, varying the key components in order to determine what experimental factors play a role in setting the color(s) of the materials. Major experimental synthetic variables studied included choice of metal (Ga, Al) and structural variations (primarily electronic) on the ligand backbone.

Initially we speculated that the colors arose from a) a highly colored and lower-valent Ga complex, b) an artifact of the chiral nature of the species, or c) a very small amount of a very highly-colored ligand degradation product. Our experimental work provided no evidence of a Ga(II) species at any point in any of the syntheses, indicating that the source of the color cannot be a reduced Ga(II) species. We prepared achiral as well as chiral Ga and Al species that exhibit multiple colors, and prepared Ga and Al complexes with a wide range of backbone-substituted ligands. This did not appear to be the source of the color either.

Our best hypothesis at this point involves the degradation (possible oxidative products) of the backbone of the di-immine ligand, since we observed the most intense colors when the backbone of the di-immine ligand contains an aromatic group. Experiments designed to deliberately degrade the ligand structure in the absence of the metalloid species did not produce highly-colored species. Alkyl substituted ligands gave colorless crystals in general. Depending on the exact substituents on the di-immine ligand, we could also unexpectedly produce cation/anion pairs, or simple Lewis acid/base complexes.

In summary, we can say with certainty that this new class of multicolored compounds is not due to differences in the chiral nature of the central metalloid, which was a motivating factor in our study, as chiral compounds can be used in nonlinear optical applications. Further study to determine the exact nature of the ligand decomposition products will be necessary.

Significance

This project has led to several conclusions that are applicable to the general area of materials science, and the specific area of chiral main group compounds. We showed that these multicolored species can be prepared with a range of ligands and metals other than gallium. However, the colors do not appear to be a result of any chiral nature of the crystal or a reduced gallium or aluminum species, but rather appear to be due to trace amounts of ligand degradation products that are highly colored.

The complexes that gave the most intense colors all used aromatic groups as part of the di-immine backbone. It is likely that small amounts of these aromatic compounds are decomposing, forming highly colored species, and then incorporating into the growing crystal lattice of the main group complex. There is enough of the highly colored impurity to alter the colors yet not enough to grossly affect the crystal structure of the complexes.

This study provides the basis for future research to understand the effects of minute levels of ligand impurities on the colors and properties of single crystals of main group compounds.

Other Communications

R.A. Kemp and A.M. Felix, “Reactions of Group 13 Compounds with Di-immines,” presented (poster) at the 2006 Gordon Research Conference, Waterville, ME, July 2006.

Molten Salt-Based Growth of Large-Area, High-Quality, Bulk Gallium Nitride for Substrates

99407

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Project Purpose

Gallium nitride (GaN) is an exciting material with the potential to enable many new products for green, blue, and ultraviolet (UV) optoelectronics and high-power, high-temperature, high-frequency electronic devices for a myriad of important technological applications. Its direct band gap is in the near-UV (3.4eV) region of the electromagnetic spectrum, is chemically stable, and exhibits high hardness.

This valuable combination of properties makes GaN unique and ideally suited for numerous important national security applications, such as chemical-biological threat detection sensors, solid-state lighting, quantum computing, more efficient UV sources for polymer curing and medical applications, nonline-of-sight communications, missile plume detection, synthetic aperture radar, and hybrid and electric vehicles.

The material has been the subject of intensive research over the past decade, and full exploitation of its capabilities will depend upon the resolution of a number of important materials challenges. Perhaps the greatest and most pervasive of challenges is the lack of availability of large-area, low-dislocation density native substrates for epitaxial growth of thin film GaN-based materials.

Current technology employs highly lattice mismatched sapphire or silicon carbide as substrates, with resulting dislocation densities in the $10^9/cm^2$ to $10^{11}/cm^2$ range. For long-lived, high-power commercial devices, dislocation densities should be on the order of $10^5/cm^2$ or less. Melt-based approaches are impractical as the required pressures exceed 4,000 to 45,000 atmospheres, and the kinetics are slow.

The successful bulk III-nitride growth approach will be one that is scalable, manufacturable, cost effective, and controllable; yields a low impurity content and high growth rate; and, of course, results in crystals of superior crystalline quality (in this case, a dislocation density below $10^5/cm^2$).

The purpose of the project is to create a bulk crystal growth technique that circumvents the difficulties presented by melt-based approaches by using a solvent, in particular a molten halide salt, which provides an excellent host environment for the GaN precursors and is liquid at atmospheric pressure in the desired growth temperature range. In particular, molten halide salts can solubilize both gallium and nitride ions without reacting with them to the extent that they are no longer available for reaction with each other. Molten salts are compatible with the 350-1200 °C temperatures needed to grow high-quality single crystal III-nitrides.

Since these molten salt-based processes can be used at or close to atmospheric pressure, scalability is not a problem, and manufacturability issues are minimized. Finally, the molten salts offer a number of pathways to improve the solubility and control the growth of the III-nitrides by functioning as an electrolyte in electrochemical processes. Unlike any existing approach, this technique offers the possibility for boule growth by virtue of two unique advantages: first, the process takes place at atmospheric pressure; and second, the precursors may be produced continuously by electrochemical methods in the reaction vessel for virtually as long as can be practically desired.

FY 2006 Accomplishments

We conducted mass transport experiments to determine whether GaN could be transported in the melt and deposited at the other end, and to determine an optimum growth temperature range. Unfortunately, we encountered a number of materials compatibility problems, from nickel migration and incorporation into the growing film at 550-800 °C, to dissolution of the sapphire substrates on which the GaN seed crystals were grown. We addressed some of the problems, but others still need attention. Based on our preliminary results, the optimum growth temperature range appears to be between 550 and 700 °C.

We dissolved lithium nitride in dimethylpropyl immidizolium imide, a room temperature ionic liquid. Attempts to reduce nitrogen gas in the ionic liquid were unsuccessful and raised questions about the behavior of trace impurities in the solution.

We reduced N_2 gas in LiCl-KCl at temperatures of 450-600 °C. Attempts to reduce N_2 in NaCl were unsuccessful due to the interference of the reduction of sodium, thereby ruling out NaCl as a solvent for this growth technique.

Significance

In order to demonstrate that a molten-salt-based growth technique is viable for substrate-quality GaN and to develop it fully, there are essentially five major technical milestones that must be accomplished:

1. demonstrate that GaN will form in the molten salt
2. demonstrate the reduction of N_2 gas in the molten chloride salt
3. demonstrate mass transport of GaN in molten salt
4. demonstrate ionic precursor transport to the surface of the seed crystal and formation of wurtzite GaN
5. fine-tune the growth process and form high quality, large area bulk materials.

We accomplished the first two demonstrations in a prior LDRD project. In this project, we focused on the third milestone. Our results indicate that it is likely this can and will happen. We desposited approximately 100 microns of a GaNi alloy (instead of GaN) on a 3 micron thick GaN film. This is significant because it shows that gallium was transported in the molten salt, lending credence to the idea of ionic and/or molecular transport in the system. The nickel was used as a support structure to suspend the spinning substrate in the melt, and it diffused into the growing GaN film.

We also made progress toward the fourth milestone. We tested the creation of the ionic precursors in the molten salts and ionic liquids at various temperatures to determine the optimum solvent/electrolyte system.

Quantification of False Positive Reduction in Nucleic Acid Purification on Hemorrhagic Fever DNA

99864

C. D. James, K. R. Pohl, K. Achyuthan, J. L. McClain

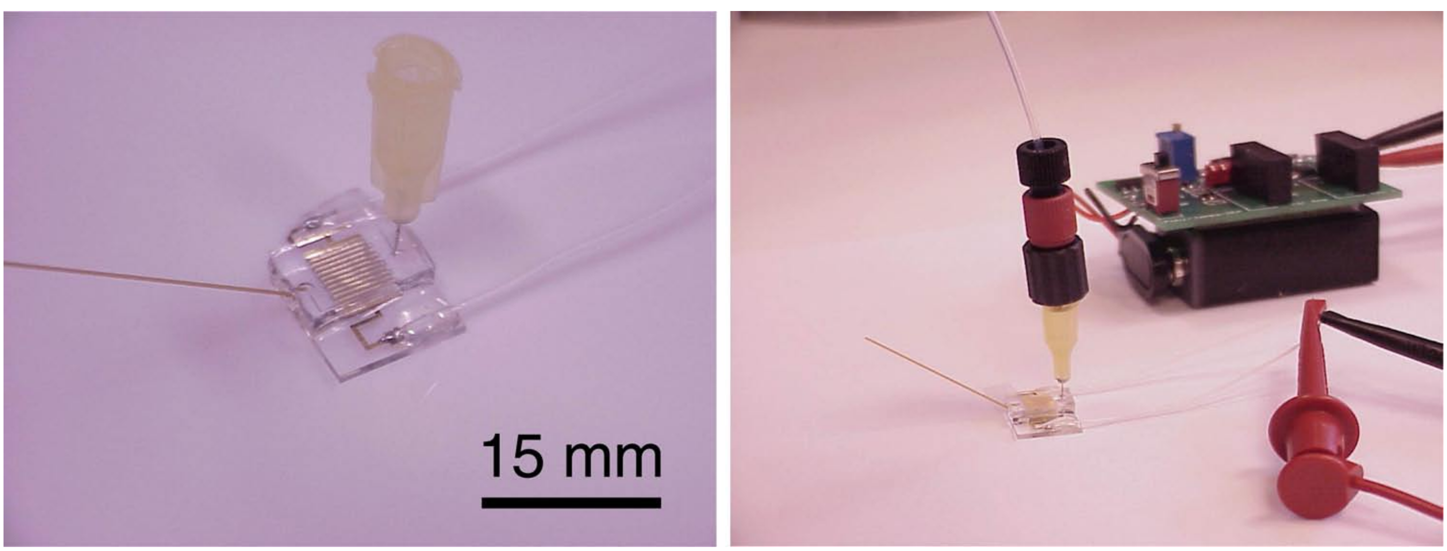
Project Purpose

Columbia University has developed a sensitive, highly multiplexed system for genetic identification of nucleic acid targets. The method has several potential applications, including clinical microbiology, biodefense, and forensics. As it exists the method is flawed due to an unacceptable rate of false positive detections. We have an invention that may fix the problem. Our invention is based on a strategy for enriching a reporter template for numerous gene amplification methods such as polymerase chain reaction (PCR) methods.

A reporter oligonucleotide binds to a target deoxyribonucleia acid (DNA) molecule, serving as a beacon for initiating gene amplification of the target. The primary obstacle to implementing this technology is the high rate of false positives due to high levels of unbound reporters that remain within the system after hybridization. The ability to distinguish between free reporters and reporters bound to targets limits the use of this technology.

In a previous LDRD project, we demonstrated several basic components of a new electrokinetic method for binary separation of kilo base pair long DNA molecules and oligonucleotides. We used static experimental conditions in that the DNA preconcentration was performed from a static droplet of sample placed on a microelectrode chip. The samples were homogeneous, and we demonstrated preconcentration of target DNA in one sample and no preconcentration on oligoneucleotides in a separate sample.

The purpose of this project was to further develop the technique and hardware for field use. Specifically, our objective was to implement separation in a heterogeneous sample (containing target DNA and background oligonucleotides), to perform the separation in a flow-based device (permitting sample introduction and processed sample capture), and to develop the components necessary for field testing a breadboard prototype system.



Assembled binary separation device with dispenser tip inlet and capillary outlet (left). Device during operation while connected to a syringe pump and the function generator (right).

The first objective is to spatially separate unbound oligonucleotides from target DNA bound to oligonucleotides. Many laboratories use dielectrophoresis (DEP) to preconcentrate DNA, but we are aiming to be the first to demonstrate a functional separation of two sizes of DNA in a mixed sample.

The second objective of implementing the separation in a microfluidic channel is important for making the device practical for sample preparation and subsequent analysis with additional techniques (PCR, and so on). The channel format enables the sample to be injected into the device and processed at the microelectrode chip, then the effluent is released from the channel and captured for subsequent analysis. In addition, the microfluidic channel has a serpentine configuration that provides longer interaction times between the sample and the microelectrode chip, thus enhancing the separation effect.

The third objective is to develop a breadboard system containing the microelectrode chip, the microfluidic channel, the megahertz power supply, and the fluid pump. The fully integrated system will allow us to test the system in the field, starting at Columbia University.

FY 2006 Accomplishments

We developed a breadboard prototype containing three crucial components: the microelectrode chip for generating the electrokinetic force for processing the sample; the microfluidic channel for enhancing the interaction time between the sample and the microelectrode chip and creating a time/spatial separation of the target DNA and background oligonucleotides; and the electronics for generating the MHz frequency voltage used to produce the electrokinetic force.

We developed a small function generator, capable of producing 10 volts peak-to-peak, with a tunable frequency ranging from 1.0 to 10 MHz. Also included in the component is the capability to produce to signals 180º out-of-phase with each other. As demonstrated in simulation and experiment in our previous project, using such a dual phase increases the strength of the DEP force, and thus the separation efficiency.

The largest component of the system as it stands is the syringe pump used to inject the sample through the device. For a related project, we demonstrated a small and compact diaphragm pump that could be implemented in this system. The primary limitation to the current diaphragm pump is that low flow rates (< 10 microliters/minute) are not possible as the pump is currently configured. With optimization of such a small foot-print pump, the entire system will fit within the size of a brick.

A second key accomplishment was incorporating surface modification techniques that significantly reduced nonspecific adsorption of the sample to the device (primarily the elastomeric channel walls). We are using Pluronic F68, a difunctional block copolymer surfactant that terminates in primary hydroxyl groups. When this surfactant is adsorbed to the microfluidic device, the hydrophobic surface groups of the elastomer polydimethylsiloxane are covered with the hydrophilic moieties of the surfactant. This produced two benefits: the channel fills at lower pressures and flow rates due to the increased hydrophilicity, and the nonspecific adsorption of DNA and oligonucleotides to the channel walls is reduced.

In summary, we succeeded in separating and preconcentrating target DNA and reduced the concentration of background oligonucleotides. Concentration factors of greater than 3 X were demonstrated for target DNA with no preconcentration of the background oligonucleotides, but reproducibility was not achieved.

Significance

Combined with Columbia University's expertise in nucleic acid identification, this technology would enable a unique, fast, and potentially compact method for detecting/identifying genetically modified organisms and multiplexed rapid nucleic acid identification.

Another separation approach is the Defense Advanced Research Projects Agency-funded IRIS Pharmaceutical TIGER platform that requires many hours and an $800K piece of equipment that fills a room. The Columbia/Sandia system could provide a result in approximately one hour, cost a few thousand dollars for the platform, and would be the size of a shoebox or smaller. This work could lead to a solution for a major hurdle in miniaturizing this biodetection system into a portable moderate-cost platform.

Other technologies such as electrophoretic separation can also be used for this type of separation. However, electrophoresis relies on direct current fields and requires large vented electrodes to apply the field and prevent electrolysis bubbles from entering the microfluidic device. In addition, high voltages are required to generate large enough fields over the separation channel distance. A final drawback to electrophoresis is that this method relies on differences in electrophoretic mobility. Thus, all analytes in the sample are subject to the electrophoretic force, and separation is accomplished only when there are differences in mobility between different analytes.

Our method uses alternating current field DEP, and since the voltages are megahertz and moderate amplitude (< 10 V peak-to-peak), there are no electrochemical affects to produce bubbles or changes in pH. This allows the microfluidic system to be completely enclosed with no venting, thus making the system safer and more robust for work with high-level biological samples as a hand-held system.

DEP is ideal for binary separations in which one analyte is subjected to the DEP force, and the second analyte is not subject to the DEP force (or more accurately, much less subjected to the DEP force). This leads to preconcentration of one analyte, while the other analyte is flushed through the system, enabling much stronger spatial separation of the two analytes.

Our results highlight the potential of this technology for genetic identification of nucleic acid targets. Several successful experiments illustrated the concept, but lack of reproducibility limited the fielding of the system. With additional research and development, we believe these technical difficulties could be overcome.

This work presents a technology to be used in mitigating infectious disease threats from a global perspective. This is an especially vulnerable aspect of homeland security due to the high level of traffic across our borders. This biodetection technology could be used in a clinical environment for screening patients, and it could be applied in a biodefense configuration for environmental, food, and livestock surveillance.

Large Atmospheric Explosions on Earth

99868

M. B. Boslough, D. A. Crawford, D. Rogers, B. N. Wylie

Project Purpose

The purpose of this project was to determine if an atmospheric explosion from an impacting asteroid could be capable of ablating the surface layer of the Earth and generating a melt sheet outside of any impact crater that is formed, providing support for the hypothesis that the Libyan Desert Glass (LDG) was formed by this process. This project also took advantage of a rare opportunity to visit the site of the LDG with colleagues who are international experts on the subject.

Most natural glasses are volcanic in origin and have chemical compositions consistent with equilibrium fractional melting. The rare exceptions are tektites, which are formed by shock melting associated with the hypervelocity impact of a comet or asteroid. LDG does not fall into either category and has baffled scientists since its discovery by British explorers in 1932.

The 1994 collision of Comet Shoemaker-Levy 9 with Jupiter provided Sandia with a unique opportunity to model a hypervelocity atmospheric impact. Insights gained from those simulations and astronomical observations of the actual event led us to a deeper understanding of the geologic process of impacts on Earth and presented a likely scenario for the formation of LDG.

We proposed that collisions of this magnitude with the Earth's atmosphere are plume-forming events. We argued that such collisions can partition the majority of the impactor kinetic energy into the atmosphere, as opposed to crater formation. By analogy with the 1994 impact of comet Shoemaker-Levy 9 with Jupiter, we suggested that plume collapse can have significant thermal effects. Because atmospheric effects are transient, such explosions tend not be recorded in the historic or geologic record. Exceptions are the 1908 Tunguska (Siberia) event, and possibly the 28.5-million-year-old LDG event.

FY 2006 Accomplishments

We ran simulations of large atmospheric explosions from asteroid impacts on the Earth with kinetic energies in the megaton to hundred-megaton size range. We simulated the airburst and impact of a 120-meter diameter stony asteroid. Ablated meteoritic vapor mixes with the atmosphere to form an opaque fireball with a temperature of thousands of degrees. The hot vapor cloud expands to a diameter of 10 km within seconds, remaining in contact with the surface, with velocities of several 100 m/s.

We were able to show that under these conditions, there is strong coupling of thermal radiation to the ground, and efficient ablation of the resulting melt by the high-velocity shear flow. Thus, we simulated a scenario that creates the conditions that could have formed the LDG.

Significance

The frequency of impacts in this magnitude range varies from a few decades to a few thousand years. The likelihood of such an event happening in our lifetime is significant and could result in widespread metropolitan-scale damage, or could be mistaken for an atmospheric nuclear test or even a first strike. There are clear national security implications, and hydrocodes used for weapons effects simulations are the most applicable to understanding this problem. Moreover, understanding the effects of such large explosions provides "ground truth" for our computational capabilities.

The risk to humans from such impacts is small but not negligible. Because of the low frequency of these events, the probability and consequences are both difficult to determine. The most likely scenario that would cause damage and casualties would not be a crater-forming impact but a large aerial burst similar to the one that created LDG.

This research is forcing risk assessments to recognize and account for the process of large aerial bursts. This work is now being leveraged and extended by a Computer Science Research Foundation project, to explore novel concepts in asteroid defense, involving nuclear explosions and determining momentum coupling coefficients for various asteroid types and explosion parameters.

Additional benefits of this project included 1) the inclusion of Sandia's simulations in a high-visibility television documentary (BBC and National Geographic) that showcased Sandia's computational capability; 2) significant and publishable advance in an unsolved scientific problem; 3) pushing Red Storm to a new range of performance; and 4) use of Sandia code (CTH) and computational facility for a national security problem (characterizing the impact threat).

Other Communications

M.B. Boslough, "Numerical Modeling of Aerial Bursts and Ablation Melting," Sandia Report SAND 2006-4263C, Albuquerque, NM, September 2006.

M.B. Boslough, "2D Red Storm Simulations of Libyan Desert Glass Impact," Sandia Report SAND2006-1068P, Albuquerque, NM, February 2006.

M.B. Boslough, "Numerical Modeling of Aerial Bursts and Ablation Melting of Libyan Desert Glass," presented at the First DTRA Hypervelocity Workshop, Philadelphia, PA, October 2006.

M.B. Boslough, "The Libyan Desert Glass: How Did It Form?" presented at the Ahrens Symposium Planetary Impacts and Physics of Planetary Interiors, Pasadena CA, May 2006.

Back-End Verification of SOC Devices

100738

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Project Purpose

As application specific integrated circuits (ASICs) move into the system-on-chip (SoC) arena, significant challenges arise. As technologies shrink to smaller dimensions, and more functionality is condensed into each design, unique functional and physical design errors appear. Faster and more complex clocking schemes compound the challenge by creating additional problems. The ASIC development within Sandia's 0.35 µm silicon-on-insulator (SOI) technology (CMOS7) must deal with these issues as well.

A few years ago, Sandia's standard design flow and tool suite could easily accommodate digital systems composed of 100,000 transistors (25,000 equivalent logic gates) and a 10 MHz clock. Today's SoC designs are typically multimillion transistors with high-speed clocks. The process phases most affected in the jump to SoC are silicon validation and the back-end physical design process; specifically, physical verification and static timing analysis. To be successful, today's design, test, and failure analysis teams dealing with these complex architectures must focus on improved design processes and better usage of the tool suite available.

The goal of this project is to develop new techniques for the back-end physical verification and silicon validation flows of complex integrated circuits. Included in our scope is the development of failure analysis methods, internal die node monitoring, and other techniques to evaluate state-of-the-art custom ASICs in a 0.35 µm SOI process in both pre- and post-silicon phases. The test vehicle is a radiation-hardened microprocessor based on Intel Corporation's Pentium I design. The results will help us evaluate current and future SoC ASICs (MESASAR, KDP) devices.

FY 2006 Accomplishments

Integrated the SEM and IDS 10K e-beam probe station into an SoC validation flow

We proved that SoC devices can be probed for functional and timing errors. Using the scanning electron microscope/IDS 10K e-beam prober, we created timing waveforms collected from a CMOS7 device and compared them with a static timing analysis model, which allowed us to validate the timing models of CMOS7 custom designs. The results will assist with future CMOS7 ASIC production.

Incorporated the FIB machine to debug and fix errors

Creating a fix or probe point can be difficult on a densely compacted device. We successfully integrated the focused ion beam machine into the debug flow of an SoC 0.35 µm SOI ASIC and used it to fix functional errors and create probe points to see into the device.

Incorporated other mechanisms to assist in silicon validation

The silicon navigation tool we evaluated allowed us to synchronize schematic and layout database views to an e-beam probe image. This accelerates debug time on a CMOS7 device and streamlines the process of finding a given trace in the metal layer definition by identifying the net in a schematic and allowing the navigational software to track the trace. We evaluated NEXS software for this capability, and there are other navigational products to be investigated. We accomplished physical verification by using a stable layer definition and repairing comparison errors between the schematic and physical design database.

Significance

The results of our project will:

- benefit the DOE mission in nuclear weapons stewardship and nonproliferation
- improve our microelectronics development process
- improve our methodology for physical design and verification of SoC ASICs
- enhance the probability of achieving first silicon functionality
- improve the reliability of the end product
- benefit upcoming SoC ASICs (MESASAR and KDP3).

Microsystem Miniaturization of High-Frequency Systems

101118

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Project Purpose

High-frequency radio frequency (RF) systems are just entering the ultraminiaturization phase that digital systems have undergone over the last three decades. The need for well-defined impedances, feature size comparable to the signal wavelength, lack of accurate software simulation for complex circuits, and radiation losses have slowed progress. However, recent Sandia progress in gallium nitride (GaN) growth science and device design, silicon germanium (SiGe) high-density RF design rules, and short channel (0.130 micron) CMOS (complementary metal-oxide semiconductor) design has enabled an attempt at miniaturizing and extending capability of complex RF systems.

The high-frequency design, computer simulation, materials, packaging, and test capabilities we develop will enable the next generation of radar systems for:

- intelligence: microtag/locators and unattended ground sensors
- satellite: large phased array radar in space for verification, high-pixel sensor systems
- Department of Defense: synthetic aperture radar (SAR) for microautonomous planes, munitions guidance, smart radio
- other government systems

The main purpose of this project was to extend Sandia's microsystems technique to accomplish an order of magnitude size reduction and performance improvement beyond our advanced SAR systems. To that end, we focused on three major areas:

The design and fabrication of the QDWS (quadrature digital waveform synthesizer) ASIC (application specific integrated circuit)

The QDWS ASIC was a "drop-in" replacement for the QDDS (direct digital synthesizer) field programmable gate array (FPGA) in the existing SAR system. In addition to duplicating the functionality of the QDDS FPGA, we designed the QDWS ASIC to integrated two banks of 128K x 36-bit error correction SRAM (static random-access memory?) on chip, as well as to lower the overall power consumption of the QDWS board in the miniSAR system.

The design and fabrication of an integrated circuit (IC) approach to an existing stable local oscillator (STALO)

This IC involved the use of a current SiGe technology available at IBM's trusted foundry. The purpose was to design and fabricate high-frequency amplifiers and surface acoustical wave (SAW) devices for inclusion in the STALO circuit.

Establish a way to fabricate GaN monolithic microwave integrated circuits (MMIC) on a silicon carbide (SiC) substrate.

Before we could design and fabricate this device, we had to develop several passive elements of the MMIC, including capacitors, resistors, inductors, and transmission lines, that were compatible with GaN and SiC. Models of these passive elements and field effect transistor will be developed for use with the microwave design of the MMIC.

FY 2006 Accomplishments

In the area of digital design:

- We set up the infrastructure with IBM's trusted foundry for fabrication of high-speed, high-density CMOS IC.
- We designed a "drop-in" replacement for the QDDS FPGA for use in an advanced Sandia SAR system.
- We characterized the phase-locked loop (PLL) and delay-locked loop (DLL) design test chips, which are part of the QDWS, and results showed better performance than the simulation goals. We designed and fabricated a QDWS IC that measures 10.6 mm by 10.6 mm and has

approximately 4M 2-input NAND equivalent gates, 5 MBit of memory, and an estimated power dissipation of 2.6 watts
- We ordered, for testing and characterizing, a package that could handle this IC and all of the inputs and outputs.

The STALO design had similar breakthroughs that gave Sandia experience in SiGe design. Again, we worked with IBM's trusted foundry on the fabrication of these circuits. We proved the STALO with a discrete benchtop prototype and implemented it with SiGe and SAW devices. The first design, verified at 3.6 GHz, showed low performance due to low gain in the SiGe transistors that was not predicted in the IBM models.

We generated and developed new, in-house models and used them in the redesign of these SiGe amplifiers and submitted them for a second IBM run. The SAW filters needed for the STALO were developed and fabricated in Sandia's CSRL and are considered leading-edge technology for microwave frequency applications.

The MMIC power amplifier showed several accomplishments in the area of GaN process development.
- We developed, characterized, and modeled passive elements like capacitors, resistors, inductors, and transmission lines for implementation on a SiC substrate.
- We developed a via-hole process that was very difficult to achieve on a SiC substrate due to the high bond energy between silicon and carbon. The vias were electrically measured and modeled for use in the design of the GaN MMIC.
- We modeled the ferroelectric transistors (FET) and chose a 900-micron device as the basic building block.
- We developed the FET process. This was difficult to achieve; we made several iterations in the process to stabilize the device electrical parameters.
- In order to allow microstrip transmission lines on the MMIC, we developed a special technique for the lapping and thinning process.
- We designed, fabricated, and characterized the MMIC.

Significance

This effort will benefit Sandia's advanced microsystems in the following areas: 1) critical technologies for next-generation fuses, 2) SARs, and 3) self-monitoring and other concepts.

Advanced RF technologies are applicable to next-generation Sandia systems and would enhance cost reduction, volume, reliability, performance, and jammer resistance. Advanced radar-enabled guidance in all-weather, global positioning system-limited environments is still a key element of Sandia's future roadmaps. We envision that initial concept verification could occur on conventional munitions using established miniSAR program contacts (Air Force Research Laboratory, Boeing, and so on).

Extreme digital/RF miniaturization is important to implementing the advanced communication circuitry needed for future concepts. For example, GaN solid-state RF circuitry will be needed to meet transmitter power requirements. Regardless of the application, future requirements will demand extreme miniaturization and performance beyond Sandia's current state-of-the-art capabilities.

We learned that weather blinding is a crippling problem for small unmanned aerial vehicles (UAVs), even at short ranges. The larger platforms provide impressive capabilities, but lack persistence. The advanced radar electronic technologies in this project would resolve these issues, making high-performance radar small and affordable while adding previously unrealizable capabilities.

Given the ever-increasing forecasts of radar and weapon needs, the potential benefits of this research are easily several orders of magnitude beyond the initial development costs. GaN is the best solution for the advanced microsystem RF concepts. The combination of high-voltage operation and high power density GaN is unmatched among semiconductors and is approximately seven times better than GaAs or Si.

Sandia will leverage existing investments in hybrid, high-power solid-state power amplifiers into MMICs for further high-frequency performance enhancements in common microsystems platforms.

Our GaN MMIC technology will help enable applications from the lower S-band to the growing attractive Ku-band. The high-speed digital circuit design of the QDWS will allow us to generate complex waveforms for many microsystems in our future. This project will benefit the development of a long-standing RF transistor technology that can be a legacy technology for the foreseeable future.

Refereed Communications

R.J. Shul, M.E. Overberg, A.G. Baca, C. Sanchez, J. Stevens, L. Voss, K. Ip, S.J. Pearton, M. Martinez, M.G. Armendariz, and G.A. Wouters, "SiC Via Fabrication and Integration for Wide Band Gap HEMT/MMIC Devices," to be published in *Journal of the Electrochemical Society*.

Integrating Nanoenabled Systems

101297

S. J. Hearne, D. L. Adams, F. T. Mendenhall

Project Purpose

The purpose of this project is to assemble a functioning embedded wireless sensor from currently available technology that will allow the sensor, memory, computing, and power source to be fabricated discretely and assembled into a single unit. This flexibility will provide a process for determining which technologies are compatible and will provide the greatest possible lifetime of the device. Using the knowledge gained in this work, we will develop a three-year roadmap for using parallel processing techniques to fabricate similar devices with greater power budgets.

FY 2006 Accomplishments

Using serial fabrication techniques, we demonstrated devices of less than 2 mm^3 that were able to sense, record, and transmit data. We fabricated the devices using commercially available components and a nanoenabled battery system. The system demonstrated a lifetime in excess 100 minutes operated solely on the available power from the battery.

Significance

This project has led to the development of a roadmap for the fabrication of an integrated system capable of sensing, storing, and transmitting data in a discrete device of less than 1 mm^3. The roadmap details a process flow to allow for the parallel processing of hundreds of devices at a time to minimize cost and provide for statistically significant numbers of devices for field testing.

Nanostructured devices will enable the deployment of embedded wireless sensors into a myriad of biological, manufacturing, and environmental applications, such as in vivo sensors with minimal soft tissue damage, and environmental analysis of biological entities where conventional analysis is impractical or impossible.

Science-Based Engineering of a Sample Preparation Device for Biological Agent Detection

102593

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Project Purpose

The development of microfluidic devices is often done by the build and test paradigm. We are demonstrating the benefits of a paradigm shift to science-based engineering by rapidly developing a sophisticated design for microfluidic sample preparation that outperforms existing technology. Mixing of samples with reagents is one of the most time-consuming operations for chemical/biological agent detection on microfluidic platforms. Based on a novel, electrokinetic phenomenon, called induced-charge-electro-osmosis (ICEO), our designs can mix a fixed volume of liquids without dispersion, a common problem with other approaches. The flow is driven by an alternating current electric field that acts on the ionic charge it induces on polarizable (metal) materials. It requires no moving parts and allows for on-chip integration. With two pairs of electrodes the field can be made to rotate, turning the flow field with it, such that it sweeps out 'dead' spots that usually exist in stationary flows. The polarizable regions can have a floating potential or an applied potential to produce different effects.

FY 2006 Accomplishments

We developed designs for microfluidic sample mixing on microfluidic platforms based on ICEO using a science-based engineering transformation (SBET) approach, where modeling was used to rapidly prototype a wide range of designs and experiments were used to validate the model and identify areas where it does not capture the physics. In partnership with the University of Louisville, along with facilities at Cornell University, we developed the microfabrication processes that can make devices according to our designs. We used a finite element model to calculate the electric field, fluid flow driven by ICEO, and mass transport in a multispecies liquid using Sandia parallel computational tools. Designs were generated that can mix in a continuous flow mode or in a batch mode. These devices should perform as well as or better than devices published in the literature. A distinguishing strength provided in batch mode is the ability to mix fixed volumes of liquids without dispersion. We coated the posts in an existing chip with gold for use in our experiments. Particle-image-velocimetry was used in the experiments to measure ICEO flows. The flow speed was found to increase quadratically with voltage, in agreement with the model. The overall flow structure found with flow visualization agreed with the model. The existing model was found to over-predict flow speeds, but the electric field strength can be increased to compensate for this and obtain the predicted device performance.

We developed a novel process for making our devices with vertical sidewalls having conductive metal coatings. The key step is ion milling, conducted at Cornell University, which preferentially removes metal on horizontal surfaces but not sidewalls. The process was used to fabricate a number of example devices with large-aspect-ratio features and is currently being used to fabricate the mixing designs generated with the modeling.

Significance

Our project is the first, to our knowledge, to design a sophisticated engineered device that harnesses induced charge electrophoresis. It makes use of specially shaped conducting posts within a microchannel and embedded electrodes for applying an alternating (AC) rotating electric field. This work benefits DOE and Department of Homeland Security needs for chemical/biological agent detection by converting some recent research into innovative technology for rapid, well-characterized, and repeatable active mixing for microfluidic applications. This will be accomplished while demonstrating the advantages of science-based engineering, a Sandia initiative. Also, this project supports the strategies of the President's American Competitiveness Initiative through participation of students at the University of Louisville and the University of California at Berkeley.

RF MEMS Passive Demodulating Detector

102597
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Project Purpose

Passive devices that require no power other than that from environmental excitation are of particular interest to several Sandia missions. For example, devices that operate over extended periods of time without a need for a battery are useful for embedded surveillance applications and structural health monitoring. To pursue this challenge, microsystem devices such as latching shock sensors and surface acoustic wave correlators have been investigated at Sandia. These devices do not require any power and operate when stimulated by an external signal (shock or radio waves).

Another passive device, an ultrawide-bandwidth (UWB) microwave demodulating detector, was conceived of by a Sandia microelectromechanical systems designer. The device offers promising enabling features such as large voltage gains from input microwave signals to the demodulated output signal. As a result, a greater efficiency is anticipated over current demodulators, and higher-frequency operation at lower power consumption is possible.

We took a science-based engineering approach to design a UWB microwave demodulating detector. We incorporated the physics pertinent to the device operation into a finite element model to explore device operation characteristics, performance, uncertainty, and optimization.

FY 2006 Accomplishments

The device physics enabling the passive demodulation were explored and implemented into two different finite element models. We first used a commercial finite element code commonly used in industry for multiphysics modeling of microsystems. This effort produced results based on the physics capability of the code and provided initial expectations and insight of the devices operation. We parametrically defined the model for suitability for uncertainty quantification and optimization. The second model was created using the Sandia-developed Sundance program. We added additional physics to this in-house code to incorporate additional heating and transfer mechanisms. We also performed optimization using the Sundance model. Both models confirmed the feasibility of the device performing passive demodulation.

Significance

We followed a science-based approach to determine the feasibility of a solution at a low initial investment. During this process, we identified gaps in specific microsystem modeling capabilities as further research opportunities. Both commercial and Sandia-developed software was used to produce results that demonstrate the operating capability and performance levels of the device. These results and the option for additional simulation runs may provide design insight to microsystem and project engineers desiring to further pursue the device.

Engineered Conjugated Molecule-Linked Metal Nanocrystal/ Silica Arrays for Integrated Chemical Sensor Platforms

102599

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Project Purpose

Chemical sensor applications of composite materials consisting of metallic (Pt, Au) nanocrystals (1-10 nm), self-assembled into networks linked by pi-conjugated organic ligands, are the subject of intense study. To date, the conductance or impedance of these materials has been investigated either in scanning-probe type junctions (individually controllable, but difficult to manufacture) or in self-assembling metal-ligand composite films (easily manufactured, but random or highly defective). In the latter materials, electronic responses are averaged over a large number of possible network paths, and film swelling often masks intramolecular conductance effects. Due to the weak chemical, thermal, and mechanical stability caused by the organic media between each nanocrystal (e.g., magnetic particles in polymer resin or organic monolayer capped particles), the resulting sensor systems lack reliability.

The purpose of this project is to investigate the use of novel self-assembled gold nanocrystal/silica nanostructures as ordered, rigid "scaffolds" for self-assembling organic ligand junctions. Electronic transport through the ligand molecular orbitals can, in principle, provide exquisite sensitivity to chemical coordination or binding events. Our unique approach will produce ordered, self-assembled networks of conducting ligand-metal nanocrystal junctions.

In prior research, we developed a direct synthesis method of ordered two-dimensional or three-dimensional gold nanocrystal/silica arrays with chemical, thermal, and mechanical robustness. These rigid structures allow molecular electronic transport effects to be measured in the absence of swelling-induced network resistance changes. The scale of these ordered structures (~ 500 nm) also provides a dimensional "bridge" between organic self-assembly (1 to 10 nm) and lithographically defined interconnects (~ 1 μm). Finally, these crystalline structures provide for control and investigation of nanocrystal separation distance effects on electron transport and sensing by allowing measurement of directionally anisotropic conductance along different crystal lattice directions.

FY 2006 Accomplishments

- Based on our previous method, we synthesized ordered gold nanocrystal/silica arrays with controlled particle size and interparticle spacing (milestone 1).
- We successfully developed two pathways to crosslink gold nanocrystal/silica arrays and form conjugated nanocrystal percolation networks (milestone 2).
- Initial I(V) current(voltage) characterization of films showed a very pronounced response to phosphonates (model analog to toxic molecules) (milestones 3 and 4).

Significance

A conjugated gold nanocrystal/silica sensing system will benefit many customers of Sandia, specifically those who desire a sensing system with low power, high reproducibility, high sensitivity, and the ability to be mass manufactured. It supports the laboratory mission by providing an excellent sensing platform that is in the national interest. The capability of controlled internanoparticle spacing and the fundamental understanding of electrical physics in nanoparticle arrays will help in the studies of electron transport in ordered nanoparticle arrays, providing a basis for fabrication of charge-transport-based nanoparticle sensor arrays. In addition, the cross-linking of dithiol conjugated sensing molecules establishes a new route for tuning nanoparticle dispersion, rheology, and self-assembly.

We established a new research collaboration with Rice University in molecular electronics and sensors. In addition, as a result of this work, we enhanced our fundamental understanding of the fabrication of nanocrystal/silica films which has benefited a current CRADA (Cooperative Research and Development Agreement) project with Lockheed Martin.

When proposed, we felt this project would serve as an excellent educational “platform” for introducing new students to the field of nanoscience. Successfully recruiting new students into nanoscience depends on our ability to quickly establish the importance of the research, aim for realistically attainable results, and unambiguously demonstrate impact of the nanoscale features of the research on device performance.

With a clear objective, a straightforward multidisciplinary research plan, a variety of research activities at appropriate levels for visiting high school, undergraduate, and graduate students, well-defined testing methodologies and success metrics, this project addressed all of these educational needs. In this respect, the project exceeded our expectations. Significant portions of the project were performed by students, with tremendous positive impact on both the project and the students, who have gained valuable experience applying nanoscience skills such as synthesis, processing, and characterization to a real-world problem.

Nanoengineering for Solid-State Lighting

102600
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Project Purpose

The purpose of this project is to develop nanoengineered solutions for energy-efficient solid-state lighting (SSL). This project initiates a partnership in nanoengineering for SSL with faculty and students at Rensselaer Polytechnic Institute (RPI). Our collaborative effort focuses on developing and applying innovative concepts to achieving energy-efficient SSL by enhancing optical efficiency of light-emitting diode (LED) materials.

Our project scope is divided into two main thrusts. The first thrust applies nanoscale engineering of dielectric materials and integration with GaN-based LED heterostructures to achieve enhanced light extraction. Our approach involves both experimental and theoretical efforts to enable effective engineering of the dielectric materials' properties in three dimensions.

The second thrust involves the study and manipulation of nanoscale InGaN materials properties to fundamentally improve internal quantum efficiency of light emitting materials. While these studies are generally applicable to all InGaN alloys, we have a particular goal of developing enhanced efficiency from InGaN alloys at deep green wavelengths where high-efficiency LEDs have never been demonstrated.

This project will effectively utilize Microsystems and Engineering Sciences Applications (MESA) facilities and will involve top-rated students in the field of nanoengineering and its application to important national problems. The new nanoengineering approaches to SSL developed here are relevant to DOE's Basic Energy Sciences (BES) and Energy Efficiency and Renewable Energy (EERE) programs and are applicable to ultraviolet (UV) light emitters for chemical-biological detection as well as high efficiency photovoltaics.

FY 2006 Accomplishments

We developed a process to achieve graded refractive index structures through a nanoscale deposition technique that is specifically tailored to InGaN-based visible LED structures. Theoretical modeling was applied to identify the most effective refractive index profiles for enhancing light extraction. This modeling effort quantified the importance of the smoothness of refractive angle within the structure and further studied the effectiveness of discrete refractive index layers in approximating a continuously graded structure.

We also developed photonic lattice test patterns of ~ 300 nm period in related dielectric films on InGaN quantum well (QW) structures. Luminescence imaging and angular emission measurements of such laterally patterned structures confirmed that enhanced light extraction has been achieved. Completing these milestones lays the groundwork for three-dimensional dielectric engineering and more quantitative evaluation of enhanced light extraction from InGaN LEDs.

Our InGaN nanoscience studies focused on gaining insight into the impact of nanoscale defects on InGaN optical efficiency. These efforts included the growth of InGaN QWs on GaN or InGaN underlayers enabling controllable nucleation of so-called "v-defects" on threading dislocations. Photoluminescence (PL) studies of these QW heterostructures revealed greater than a tenfold increase in PL intensity compared to InGaN QW samples without underlayers. Temperature-dependent PL verified that this luminescence enhancement is related to an increase in the internal quantum efficiency of the QWs.

Room-temperature scanning cathodoluminescence measurements revealed dark spot regions correlated with v-defects (~ 150 nm size) in addition to the presence of distinct spatial regions of enhanced PL on the micron scale. We performed temperature-dependent cathodoluminescence studies on single quantum well structures and provided quantitative assessment of the internal quantum efficiency improvements enabled by underlayers and further evidence that light enhancement was achieved in the material regions between v-defects.

These results are qualitatively consistent with a theoretical model by Hangleiter, et al. [1] that predicts that v-defects decorating threading dislocations can effectively block carriers from the dislocation, making the defect region dark while simultaneously avoiding nonradiative recombination. Notably, our combined x-ray diffraction and atomic force microscopy measurements indicate that v-defects do not decorate all dislocations in the samples and therefore may not preclude all pathways to nonradiative recombination.

To gain greater insight into recombination at undecorated threading dislocations, we developed a theoretical model to evaluate carrier recombination in the vicinity of a charged dislocation core. This model predicts a distinct carrier density dependence for recombination rates near dislocations that may explain the long-observed drop in efficiency of InGaN LEDs at high current densities.

Significance

This project aims to apply nanoscale science and engineering to enable significant energy efficiency improvements in visible LEDs for solid-state lighting. Our combined experimental and theoretical efforts can be further developed to enable greater insight into the impact of nanoscale defects on the internal quantum efficiency of InGaN based LEDs. If LED efficiency performance metrics defined in the SSL roadmap can be met, SSL could have a tremendous impact by reducing US electricity consumption by 10 percent, saving about $25B/year (by 2025).

The nitride semiconductor materials developed in this project can also be applied to other device technologies of interest to Sandia and agencies such as the Defense Advanced Research Projects Agency. These applications include InGaN photovoltaics as well as AlGaN UV LEDs and laser diodes for fluorescence-based chemical-biological sensing and water purification. The dielectric nanoengineering strategies developed in this project also have broad applicability to semiconductor optoelectronic devices and, as an example, may contribute to enhanced performance in photovoltaics.

[1] A. Hangleiter, et.al., “Suppression of Nonradiative Recombination by V-Shaped Pits in GaInN=GaN Quantum Wells Produces a Large Increase in the Light Emission Efficiency,” *Physical Review Letters*, vol. 95, p. 127402, September 2005

Miniature Vibrational Energy Harvester: Improved Modeling and Simulation Through Experimental Validation

102602

R. A. Kellogg, J. K. Brotz, H. Sumali

Project Purpose

Miniature, inexhaustible power systems are needed to enable persistent monitoring for critical national security systems. One means to satisfying this need is through the use of vibration-based miniature power generators, which extract vibration energy from the environment to produce and store electrical energy. A robust mesoscale vibrational energy harvester prototype, based on electromagnetic induction principles, has been successfully produced at Sandia. To best implement the technology, however, further gains in output performance and miniaturization are desired.

The purpose of this work is to gain further understanding and produce technological improvements in the vibrational energy harvester technology. This is being achieved through science-based engineering founded on comprehensive electromagnetic and mechanical dynamic models of the energy harvester prototype. Our interdisciplinary team has experimentally validated preliminary models and integrated this information into an advanced modeling and simulation capability for the electromagnetic vibrational energy harvester technology. These advanced models will ultimately allow the performance of vibrational energy harvesters to be accurately predicted, thus facilitating their design and operation at the microscale.

FY 2006 Accomplishments

Models of the magnetic and dynamic systems for the electromagnetic-based vibrational energy harvester were improved and validated through experimental analysis of a Sandia prototype. We completed a high-fidelity finite element model of the prototype's magnetic components to address as-fabricated variations in components and as-measured material properties. This led to refined transduction models that predict a maximum energy harvester output performance at resonance of 1.57 volts and 250 micro-watts, which is within 10 percent of that observed for the prototype. Subsequently, we performed a thorough sensitivity analysis over the magnetic design space. Key results show that small variations in magnet strength have a large impact on voltage output performance.

Using the improved transduction model, we validated an end-to-end Simulink model accounting for the mechanical and electromagnetic interactions through prototype testing. Validation experiments with laser Doppler vibrometry show the model output to be within 16 percent for sinusoid and random vibration environments. The random vibration environment for an automobile was also replicated in simulation and experiment. Experimental results were in good agreement with simulation and demonstrate energy harvester power outputs of 30 microwatts. We also conducted an analysis of the prototype's dynamic characteristics over linear and nonlinear regimes. This analysis allows for independent verification of the finite-element derived force-displacement relationship of the magnetic system.

Work of particular value for future smaller dimensionally scaled systems addresses gains in energy harvester efficiency. We successfully modeled a synchronous rectifier to explore a high-efficiency voltage rectification and multiplication scheme. Additionally, we used a vacuum test system and integrated shaker table to measure the effects of air damping and to isolate electromagnetic damping effects on performance. To guide future development of the vibrational energy harvester, we contacted US government agencies to obtain performance objectives for national security applications.

Significance

Miniature vibrational energy harvester technology will provide long-lived power supplies for mobile, remote, and autonomous national security devices. Applications include intelligence gathering, health monitoring, and passive identification of vibration environments.

Model-Based Statistical Estimation of Sandia RF Ohmic Switch Dynamic Operation from Stroboscopic, X-Ray Imaging

102603

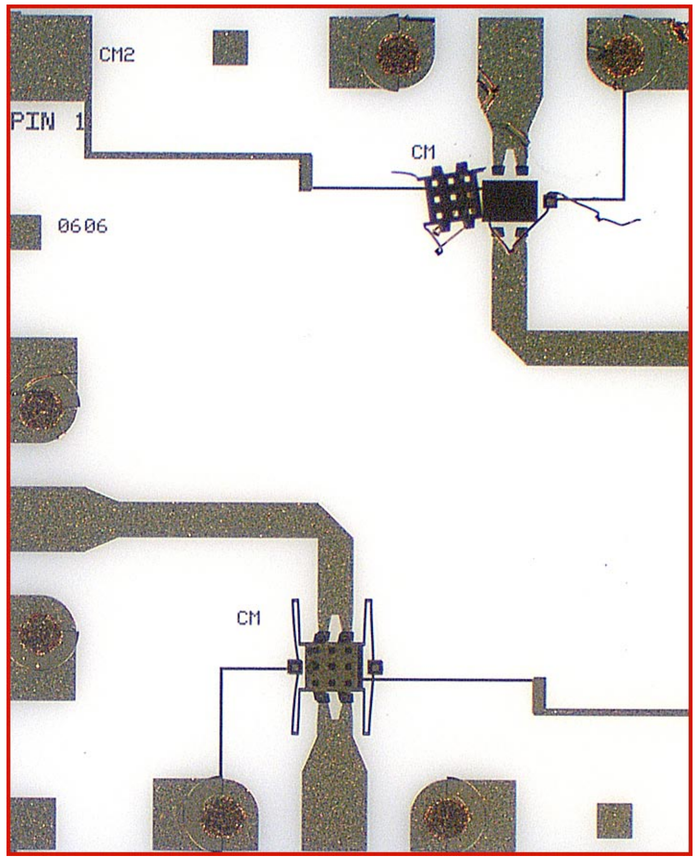
C. F. Diegert, T. J. Miller, K. R. Thompson

Project Purpose

We are developing a new experimental diagnostic for measuring dynamic operation of Sandia's radio frequency (RF) ohmic switch. The new diagnostic is enabled both by dramatic advances in the predictive capability obtained from Sandia's science-based engineering transformation (SBET) approach (applying numerical simulation tools) and by evolutionary advances in microscopic imaging using x-rays and light. Modeling and simulation will contribute to future Sandia switch designs by including the use of numerical simulation with other SBET process steps involving measurements on experimental switch parts (physical experiments).

The purpose of this project is to apply the SBET process to get a low-degree-of-freedom summary model that is useful as a basis for statistical estimation. The SBET basis for the summary model includes results from multiple simulation experiments based on large-degree-of-freedom finite element analysis (FEA) models. A successful summary model can enable understanding, validation, and other contributions from measurements of switch operation by constraining statistical estimation. For example, FEA can sort out expected motion of the RF switch plate as a superposition of a handful of mode shapes.

We are acquiring and analyzing stroboscopic, x-ray images of an operating switch and performing a statistical estimation computation to estimate the particular weighted combination of modes that best explains the observed images. Statistical estimation theory has been model-based for more than a century. However, we are attempting something new: we are making effective use of large-scale, finite element analysis as a component of our statistical estimation methodology. Our learn-by-doing approach demands physical samples of RF switch parts and highly predictive numerical solutions to FEA models of their operation. Both are readily available at Sandia as a result of investment in microsystems and in advanced simulation and computing. Our work is focused on innovation in making the physical measurements and in analyzing these measurements.



Radio frequency switch imaged on light microscope

FY 2006 Accomplishments

We designed and fabricated a test fixture suitable for observing dynamic RF ohmic switch operation on typical x-ray machines. We acquired static x-ray images of an RF ohmic switch part using a microsource x-ray machine and a nanofocus x-ray machine. We began working with vendor FEINFOCUS to obtain imaging from Sandia's nanosource machine that exceeds the resolution we obtained on the microsource machine.

We acquired surface topography of an operating RF ohmic switch from an ultraviolet-light confocal microscope. We completed careful, but conventional, analysis on a confocal image stack; the directly observed topography showed that activating this switch part produces a 400 nm bend in its plate. We performed a speckle correlation analysis of the confocal imaging that, surprisingly, shows a subpixel, in-plane deformation (movement) of the plate that indirectly implies the bending.

Significance

We showed that a computationally intensive numerical solution can be used as an integral part of making difficult, nanometer-scale measurements. This innovation breaks the constraints implied when physical measurements are cast in a formal verification and validation (V&V) role. For V&V, measurements are made independently, without incorporating high-performance solutions to FEA or other computationally intensive models. Our work shows a role for solving a large FEA problem as an integral part of making measurements.

Our results have already helped understanding deformation in Sandia's RF ohmic microelectro-mechanical systems (MEMS) switch part. The innovation is that the predictive capability of the numerical solution is assumed correct and forms a basis for measuring which dynamic deformations occur in the physical switches under test. The specific methodology, with application of x-ray imaging and speckle correlation, can contribute important measurements on a variety of operating MEMS.

We look forward to attempting measurements that contribute to understanding dynamic operation of Sandia's new thermal MEMS actuators and are complementary to measurements made using interferometry and by laser Doppler. More generally, the work opens a broad front of possibility where the ability of massive high-performance computers enables new measurements.

Integration of Nanoporous Materials into Device Structures via DEP-Directed Manipulation and Templated Self-Assembly

102604

R. Shediac, A. A. Talin, M. P. Kanouff, R. R. Stumpf, B. A. Simmons

Project Purpose

Predictable and reproducible manipulation of materials and objects at the nanoscale is one of the greatest challenges that must be surmounted to develop these materials into useful products. Nanoporous materials such as metal organic frameworks (MOFs) and carbon nanotubes (CNT) have properties that make them extremely attractive for fabrication of advanced nano- and microscale devices due to their extremely high surface area and tunable pore chemistry. Integration of nanoporous materials with such devices is challenging due to synthetic conditions incompatible with device structures and/or morphologies that are not useful for devices. Methods are needed that allow manipulation of the materials after growth is completed or enable controlled growth with minimal defects under mild conditions.

The objective of this project is to lay a foundation for the predictable and reproducible integration of nanoporous materials with micro- or nanoscale devices by developing device-compatible synthesis and/or manipulation methods. We focus on two important classes of assembly methods: 1) dielectrophoretic (DEP)-directed post-growth assembly of nanotubes/wires and 2) templated self-assembly of MOFs. Electrode arrays and microcantilever sensors will provide the test platforms. We are collaborating with Georgia Tech to leverage their nanowire tracking and modeling capabilities and their microsensor expertise.

FY 2006 Accomplishments

We calculated the electric field used for assembly of nanorods by DEP using a finite-element model. Rod trajectories were calculated using the dipole-moment method. The Clausius-Mossotti factor was modified based on the Maxwell stress tensor to improve accuracy. We obtained preliminary results for other effects, such as AC (alternating current) effects across double layers, AC electro-osmosis, and a rod surface charge. We fabricated wafers with gold electrodes and used them in experiments for comparison to the model. The model identified the region where Brownian forces exceeded DEP, impeding assembly, showing that the rod number density may have been too low in some experiments.

We achieved controlled growth of MOF crystals from self-assembled monolayers on gold substrates, providing a direct route to deposition of MOFs on prefabricated microcantilevers. Important characteristics such as the size and uniformity of MOF crystals and the degree of MOF coverage on the substrate were reproducible after optimization of synthesis parameters such as growth time, reaction temperature, and reactant concentrations.

Under these growth conditions, the individual cubic MOF crystals have dimensions of approximately 20 microns, a size which is compatible with the dimensions of the microcantilevers, suggesting that it should be feasible to nucleate a small number of MOF crystals directly on prefabricated microcantilevers. Density functional theory was used to establish models of MOF-metal oxide interfaces, providing a framework for evaluating the adhesion and stability of MOF layers on different device substrates.

Significance

The results of this project will advance our ability to control, manipulate, and integrate nanomaterials with both micro- and nanoscale devices, thereby impacting a number of critical Sandia technologies. MOFs and CNT are chemically tunable materials with exceptionally high surfaces areas whose properties have numerous device applications. If successful, these devices will impact impurity detection in water or waste streams, chemical weapon detection, and ultrasensitive gas sensors for monitoring the health of nuclear weapons.

In addition, this project supports a Sandia objective to incorporate nanoporous materials on microelectro-mechancial devices. Specifically, the challenges of scaling and reproducing synthetic approaches, integration of nano- and microsystems, and interfacing disparate materials are addressed. We are also forging a relationship between Georgia Tech, one of the nation's leading engineering schools, and Sandia in an area with wide applicability to Sandia missions.

Refereed Communications

R. Shediac, E. Lai, C. Bauer, B.A. Simmons, R.R. Stumpf, A. Choudhury, P.J. Hesketh, and M.D. Allendorf, "Growth of Metal Organic Frameworks onto Microcantilever Substrate Materials," presented at the Electrochemical Society Fall Meeting, Cancun, Mexico, October 2006.

B. Simmons, D. Bahr, M. Allendorf, W. Mook, C. Bauer, and N. Moody, "Synthesis, Characterization, and Mechanical Properties of Metal Organic Frameworks," presented at the Fall Materials Research Society Meeting, Boston, MA, November 2006.

Atomistic Simulations of Brittle Crack Growth

102605

J. J. Hoyt, E. B. Webb III, A. P. Thompson, R. Tandon

Project Purpose

Due to their many desirable properties, bulk and thin-film brittle materials are used in a large number of mission-critical components. Although these materials perform key functions in many technologies, their intrinsic lack of ductility can lead to environmentally induced, slow (< 10^{-10} m/s) failure during manufacturing, qualification, storage, or use. The necessary conditions related to the onset of fracture are very difficult to characterize experimentally because the governing chemical and physical interactions between the material and environment occur at the atomic scale. In addition, state-of-the-art continuum models are unable to properly describe fracture propagation probabilities, primarily because they are based on purely empirical correlations with piece-part fracture statistics.

The empirical-based approach to modeling results in unacceptably broad uncertainty in long-term reliability predictions and in component designs that are either overly conservative/expensive or have poor reliability. To transition from an empirical to a science-based modeling approach, an understanding of the physics of atomic scale processes is needed. In this work, large-scale molecular dynamics simulations are used to investigate the atomic structure at the crack tip in silica.

FY 2006 Accomplishments

We developed important techniques for generating molecular dynamics simulation cells of amorphous silica. In particular, we perfected a method for introducing an atomic scale crack into a bulk amorphous sample. The crack-introduction scheme maintains the 1:2 Si-O stoichiometry and allows specifying a crack length and crack opening. In addition, we determined the bond angle distribution of Si-O-Si and O-Si-O bonds from equilibrated samples in the vicinity of the crack tip and compared the results to the distributions in the bulk.

Significance

The scheme developed for generating an atomistic crack in an amorphous silica system sets the stage for further molecular dynamics studies of the threshold stress required for crack propagation. The results will improve the prediction of failure in weapons components and advance our mission of stockpile stewardship.

Integrated Machining and Assembly of MEMS-based Antenna

102606

J. A. Palmer, P. G. Clem, B. D. Chavez, B. H. Strassner II

Project Purpose

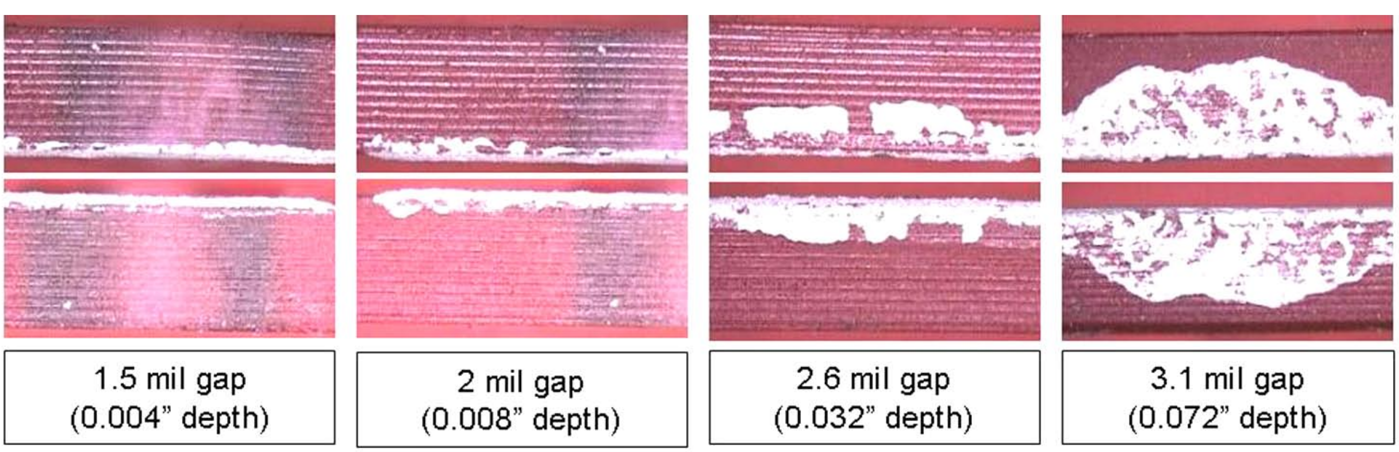
The challenge of monolithic or embedded systems is that the electrical components also serve as the mechanical interconnects and housing. In this project we are improving the mechanical and electrical characteristics of a low-profile microelectromechanical system (MEMS)-based sensor antenna using novel manufacturing techniques.

With recent advances in rapid prototyping of high-density circuitry (RPHDC), three-dimensional multimaterial structures can be fabricated with high resolution. By leveraging capabilities of Sandia, Utah State University, and the University of Texas at El Paso, we are hybridizing (colocating) stereolithography (SL), direct write (DW), and Solidica® ultrasonic consolidation (UC) fabrication processes. Until now, these approaches have not been applied to complex microwave antennas structures such as arrays or those with embedded MEMS devices. Yet, these systems significantly impact portable, unmanned aerial vehicle (UAV)- and satellite-based applications.

The combination of the SL, DW, and UC techniques not only improves placement and interconnection of embedded devices (which is extremely important at high frequencies), but creates a minimal and unitary assembly that is simpler and more amenable to electromagnetic modeling. Currently, electromagnetic modeling of an entire array system with embedded electronics is extremely difficult and slow, if possible at all. Another key advantage of the SL, DW, and UC techniques is that they encapsulate and build a unitized structure at or near room temperature; this is a necessity for embedding MEMS devices. In fact, the SL, DW, and UC techniques are well-suited to transition from the microscale of MEMS packages to the meso- and miniature-scale of other electronic components.

Successful hybridization and application of SL, DW, and UC have the potential to change how systems are designed and built with greater performance margins. Additionally, this project integrates MEMS components into a real-world system in such a way that they can be truly effective and ultimately become a pervasive high-impact technology.

Without this multidisciplinary approach, modular or embedded electronics such as tags, sensors, antennas, or optics are often unnecessarily bulky, heavy, and expensive. This is undesirable, in general, but particularly important for scenarios where large quantities are needed and units need to be considered expendable.



Samples of E1660 ink penetration depth vs. gap width.

FY 2006 Accomplishments

We have made some early discoveries as well as established a foundation for interdisciplinary science and engineering to expand the variety and understanding of these techniques for electromagnetic devices and applications. Highlights include:

- First demonstration of Solidica ultrasonic consolidation with DW of conductive ink to create antenna array interconnections.
  - The construction eliminated human and mechanical alignment errors. This should improve the electrical performance of the antenna array assembly.
  - The via interconnect was encapsulated in the UC antenna body. This should yield a monolithic construction with low losses.
- Construction of a lightweight honeycomb cage structure for the antenna array.
  - This design will mount to the existing miniSAR (synthetic aperture radar) gimbal.
  - Allows for air cooling of electronic devices.
  - The low-weight assembly will improve the balance, vibration, and mechanical control as the antenna array is steered.
- Strength and flow tests on conductive inks.
  - Permitted choice of ink based on electrical and mechanical needs.
  - One use of DW ink, providing ground connectivity from the chassis to antenna cards at the slot openings/gaps, did not require high conductivity but did require an ink that was more viscous and had larger particle sizes.
  - Another use of DW ink, providing a via interconnect between the antenna and power divider (spine) cards, required high conductivity and needed to survive a mechanical pull test.

We also submitted a technical advance (invention disclosure).

Significance

Marrying advanced manufacturing techniques with advanced electronics is essential for state-of-the-art systems with high-performance margins and real-world feasibility (cost, weight, and so on). Our interdisciplinary approach stimulates concurrent engineering both in education and in practice. Additionally, this effort aims to integrate MEMS components in such a way that they can be truly effective and ultimately become a pervasive high-impact technology in many national security applications.

Ultimately, this work strategically combined multifaceted (i.e., computational modeling and experimental validation) and multidisciplinary (i.e., electromagnetics, materials, and manufacturing) approaches for science-based engineering.

Refereed Communications

C.J. Robinson, B. Stucker, A.J. Lopes, R. Wicker, and J.A. Palmer, "Integration of Direct-Write (DW) and Ultrasonic Consolidation (UC) Technologies to Create Advanced Structures with Embedded Electrical Circuitry," presented at the Solid Freeform Fabrication Symposium, Austin, TX, August 2006.

Neutrino Detection Technology Development

102607

N. Bowden, M. Allen, J. S. Brennan, J. K. Estrada

Project Purpose

The goal of this project is to develop new concepts and technologies related to reactor antineutrino detection technology, with the ultimate goal of broadening the fieldability and application space of this new reactor monitoring technique. The fundamental design concepts and technologies underlying antineutrino detection have changed relatively little in the last few decades, suggesting that a revolutionary approach could provide large performance improvements. We will seek such advances by:

- revisiting the fundamental assumptions that are typically applied to detector design and operation, and thus to seek new design concepts utilizing existing materials
- identifying and testing new detection materials that might allow considerably more information to be gathered from particle interactions in a cost-effective and simple manner
- engaging with the academic antineutrino detection community so as to keep abreast with the current state of the art in the field.

FY 2006 Accomplishments

Much was achieved in FY 2006:

- A promising new detector design concept was developed that combines many desirable features that may allow the deployment of antineutrino detector above ground (currently, they must be buried). We took a novel approach – separating the two components of antineutrino detection (positron and neutron detection, which are typically combined) allows each to be independently optimized and allows a wider variety of detection media to be considered. The detector segmentation inherent to this separation allows for efficient particle tracking to be implemented – an important background rejection technique.
- A promising material was "rediscovered." *Trans*-Stilbene is an organic crystal scintillator that has many attractive features; however, it is little used due to anisotropies in its light emission. With careful experimental design, it might be possible to use this anisotropy to reconstruct the direction of the incident particle. This effect was observed in the 1970s but has since been little studied. We were unable to obtain the material and conduct experiments in FY 2006, but we hope to have the opportunity to do so in the near future.
- We presented and discussed our detector design concepts at two international workshops/meetings, where our input was well received, and we learned a great deal about related international efforts.

Significance

The goal of this research is to facilitate the development of a new generation of antineutrino detectors for use in monitoring plutonium production at nuclear reactors. The size and performance of the current generation of antineutrino detectors severely limits the applicability of this technique. For example, detectors must currently be located ten of meters underground, while being within a few tens of meters of the reactor core.

Design concepts, technologies, and materials that would allow above ground and/or larger standoff would greatly increase the utility and application space of this monitoring technique. Development of such technology at Sandia would ensure our continued presence at the forefront of applied neutrino physics, and would also have potential application to fundamental science measurements that use neutrino measurements.

Other Communications

N. Bowden, "Experience with Compact Antineutrino Detectors for Reactor Safeguards," presented at the Workshop on Antineutrino Detector Design, Rio de Janeiro, Brazil, 2006.

J. Lund, "Alternative Designs for Inverse Beta Detection," presented at the Applied Antineutrino Physics Workshop, Livermore, CA, 2006.

Optical Microswitching Foundations

102609

E. J. Garcia, S. N. Kempka, D. M. Tanner, M. A. Polosky, G. R. Bogart, K. A. Peterson

Project Purpose

The purpose of this project is to develop a science-based engineering (SBE) approach to create the scientific underpinnings and foundations needed to address a high-risk/high-payoff technical challenge faced by the future nuclear weapons (NW) stockpile: an integrated, high-reliability microsystem.

To meet NW strategic missions and goals and to realize the Microsystems and Engineering Science Applications (MESA) vision, the scientific foundations of optical-electrical power conversion, MEMS energy storage, and reliability and signal discriminator technologies must be developed and established to enable future microsystems insertions in NW. We are developing an SBE approach to create these scientific foundations with a MESA integration team of technical leaders in key areas of modeling/simulation, electro-optical devices, MEMS discriminators, and MEMS reliability to develop an engineering sciences design basis that crafts prior LDRD research investments and newer discoveries into a foundational capability.

SBE fuses science-based modeling and simulation, systems engineering, concurrent engineering, and weapon and component system engineering to develop advanced microsystems capabilities that would be impossible to develop using prior methodologies. We identified essential, fundamental technologies that require a profound understanding of their underlying physics:

- Energy conversion and energy storage technology
  - Scientific basis for high-reliability systems
  - Technology development
  - Modeling, simulation, and validation
- Reliable signal discriminators in the microdomain
  - Novel concepts and underlying physics
  - Modeling development and validation
  - Requirement assessments
- Advanced packaging and integration technologies
  - Scientific underpinnings
  - Process investigations
  - Modeling and simulation

We are investigating these areas based on prior related work and new investigations, both experimental and mathematical, to create physics-based predictive models that permit predictions of microsystem component performance, including aging and reliability, in a variety of environments.

FY 2006 Accomplishments

We developed eight signal discrimination concepts; the leading concept uses a counter meshing gears (CMG) mechanism to perform discrimination and is driven by nanotractors providing a significant force margin. Other concepts under investigation incorporate fiber and free-space optics ideas with switchable optical absorbers, micromirrors, and microrelays implemented through evanescent coupling, wavelength dispersive multiplexing and resonance. The nanotractor-driven CMG design is complete (16 mask design in SUMMiT™), along with a thermally actuated CMG discriminator version, and is being fabricated.

We developed a testing capability to measure functionality of CMG discriminators and nanotractors. An accelerated aging experiment (at 250 °C) was started to study hub/gear interactions and adhesive forces.

A rigid-body dynamics model of the discriminator, which also incorporates flexible-body elements, was formulated and is being used to compute system responses to normal and abnormal environments. We demonstrated a three-dimensional simulation of the transient mechanical response of a thermal actuator. The simulation includes fully coupled physics (electrical, thermal, and structural), is fully dynamic, and includes nonlinearities. We developed a nonlinear optimizer with nonlinear inequality constraints to determine optimal actuator designs that minimize input power and energy. We are modeling previously fabricated SUMMiT-based thermal actuators to correlate our models with actual hardware response.

An optoelectronic system that converts optical inputs into appropriate electrical signals, that in turn provide a code to a microelectromechanical system (MEMS) discriminator (and, when unlocked, provides power to MEMS-based optical fiber switches), has been designed and is partially fabricated. Simulation indicates the optoelectronics system will be functional; in addition, experimental data has validated some parts of the system. Separately, an extremely small MEMS drive circuit incorporating a high-voltage photovoltaic has been used to drive micromirrors with room light. We demonstrated packaging and interfacing fibers with actuators.

Significance

One of the most compelling needs for the future NW stockpile can only be addressed by an integrated all-optical discriminating microswitch. In this project we are developing a science-based engineering approach that addresses high-risk/high-payoff technical challenges associated with such a device.

The all-optical signal discriminator portion of a functional microsystem is useful by itself and must integrate optical-to-electrical power conversion and storage with a reliable signal discriminator. Over the past 10 years, Sandia has invested in high-voltage optical-electrical power conversion as well as MEMS energy storage, packaging, reliability, and signal discriminator technologies. To meet NW missions and goals and to realize the MESA vision, these foundational investigations must be fused into a packaged, functional microsystem.

A microsystems-enabled transformation of the NW stockpile requires a change from traditional weapon and component engineering development methods. Traditional methods generally emphasize testing and inspection at system and subsystem levels using mature technologies and well-known materials in a conservative design environment. This approach effectively achieves extremely high reliabilities but at high costs and over long development times.

We intend to advance the SBE approach that will form the basis for the development of microsystems under contemporary schedule and budget constraints. To this end, we assembled a multidisciplinary team composed of technical leaders in four key areas (modeling/simulation, electro-optical devices, MEMS discriminators, and MEMS integration and reliability) to develop an engineering sciences design basis that captures prior research results and crafts them into a future NW component concept. This approach will provide a foundation for improving NW safety using advanced microsystem technology.

New Processes for Innovative Microsystems Engineering with Predictive Simulation

102610

S. W. Thomas, J. E. Massad, M. S. Baker, R. A. Kellogg, C. W. Dyck

Project Purpose

The purpose of this project is to discover processes by which computational simulation might be used for creative design problem solving for an otherwise refractory multiphysics exploratory design problem. Can we use predictive simulation to help us make a nonlinear, creative leap from a design that does not work to a design that does? The particular test bed design problem we chose was the redesign of a high-speed electromechanical switch (HSEMS) that had been through several design-fabrication-test cycles, achieving promising levels of performance (switch frequency, power consumption), but failing to meet reliability requirements (average number of switch cycles before failure).

Prior to starting this project, the measured performance of the latest HSEMS design had been successfully explained using large-scale, high-performance modeling and simulation via the dynamics code Andante. Local design optimization and sensitivity analysis via Andante simulation also showed that no local, linear perturbation of the HSEMS design could sufficiently improve the reliability. Our objective was to discover how computational engineering techniques (e.g., modeling, simulation, optimization) might be extended to support derivation of a successful design that must be a nonlinear (and perhaps "creative") leap from existing designs.

FY 2006 Accomplishments

As a result of experimentation with new design models and optimization algorithms, we found a qualitatively different new HSEMS design that simulation predicts will likely meet the objective of substantially improved reliability, while meeting performance constraints. We hope that fabrication and test of this new design in FY 2007 will validate these predictions.

The computational engineering process we used is based upon 1) embedding the existing design in a substantially larger design class, allowing for qualitatively new design approaches; 2) building a parameterized simulation model for this new design class; and c) applying a new, global, computational optimization algorithm to the simulated design parameters.

Global optimization revealed local optima corresponding to design approaches that we probably would not have found with standard optimization codes. At least one of these optima corresponded to a design that is substantially different from the existing design in ways that are intuitively surprising. For example, electrostatic actuation replaces actuation via a spring retractor. Moreover, simulation predicts that the new design will meet the goal of substantially improved reliability. We believe this technique, using global optimization to find large, counterintuitive design "leaps," will be generalizable to other challenging design problems.

Significance

For a single, focused problem, this project demonstrated a new computational engineering process that appears to provide significant support for creativity in complex engineering design. We find this result significant, because it runs counter to expectations of many design engineers: that modeling and simulation are primarily (only) useful for analyzing existing designs, and perhaps, via optimization, for design refinement. To the contrary, we found that global optimization could find a radically different design, which simulation-based analysis predicts solves a design problem that has been otherwise refractory over multiple design-fabrication-test cycles.

The computational engineering steps for the new process are in principle generalizable across a broad range of DOE mission scenarios involving complex design for which innovation is a critical component. These steps are: 1) embed the (remotely) feasible class of designs in a parameterized design model; 2) couple suitable computational simulation code to this model to predict and analyze performance of an arbitrary design realization; 3) apply global optimization on one or more performance metrics to find many or all local optima of the design parameters; 4) scan the local optima and identify any that correspond to large improvements in performance and/or apparent changes in “design intent,” and 5) fully analyze the latter, with the aim of introducing new design(s) into fabrication and test.

The results of this project suggest that this process will support significant increases in design innovation and resulting design performance. Our results also suggest that algorithms for provably convergent global optimization of objective functions typical of those derived from engineering simulation are worthy of further research, development, and code implementation.

The set of such algorithms, probably convergent for “egg-carton” functions having, for example, known variograms, is very new and is to our knowledge not implemented anywhere in production codes at Sandia or DOE. The engineering process would benefit from algorithms that go beyond even current theory, for example, to provably find all the local optima with objective function values in a given range.

Embedded Evaluation Sensor

102612

M. S. Baker, R. Chanchani, R. A. Kellogg, D. S. Epp, M. E. Kipp

Project Purpose

The purpose of this project is to determine if a MEMS (microelectromechanical system) passive latching shock threshold switch can be used to detect an environment of interest to the Sandia community. The focus of the work for FY 2006 was to understand the relevant environment and predict the response of the packaged MEMS shock switch to this environment.

Once feasibility was determined in the modeled response, the device was then tested in environments with similar shock amplitude and frequency content to that of the intended application. This final test is important for feasibility demonstration, and data collected will be used in future design revisions to improve the device performance.

A substantial modeling effort was needed to understand the environment of interest and to determine if the MEMS shock switch is able to respond to this environment. This modeling work provided the input acceleration histories that are necessary for the design of the MEMS sensor, as well as the design of experiments to test feasibility.

FY 2006 Accomplishments

We accomplished all of the milestones outlined for FY 2006. The operating environment was evaluated and modeled using the Sandia developed CTH code to define the expected shock inputs to the sensor. The resulting profile has peak amplitudes of just over 10,000 g, with an average magnitude of 5,200 g over a duration of approximately 40 microseconds. We then modeled the dynamic response of the shock switch due to this predicted input acceleration for all the available MEMS switch designs (from 85 g to 12,500 g).

This device-level modeling was performed using the commercial finite-element code ANSYS. The model predicts a switch closure for all but the highest g device, as would be expected based on their static set-points.

We verified device functionality in centrifuge, drop-table, and Hopkinson bar testing. We will use the results of these experiments to validate and improve both the shock modeling and the device-level modeling of the sensor.

We identified a suitable commercial package for functionality testing at this stage. We tested this package for survivability of the ceramic package, die attach, and wire bonds up to 27,000 g. Additionally, we characterized the shock attenuation for JM7000 die attach on a shaker table up to 100 kHz input frequencies.

The testing accomplished to date demonstrates feasibility of this concept and highlights several areas that need additional development.

Significance

This LDRD project is relevant to the DOE mission of ensuring the safety and reliability of the stockpile. We contributed to the base of knowledge on MEMS performance and survivability at high shock levels with experimental data that was previously unavailable. This will be important for all future MEMS work in this field as a baseline for performance and survivability.

Phase Imprint Lithography for Large Area 3D Nanostructures

102613

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Project Purpose

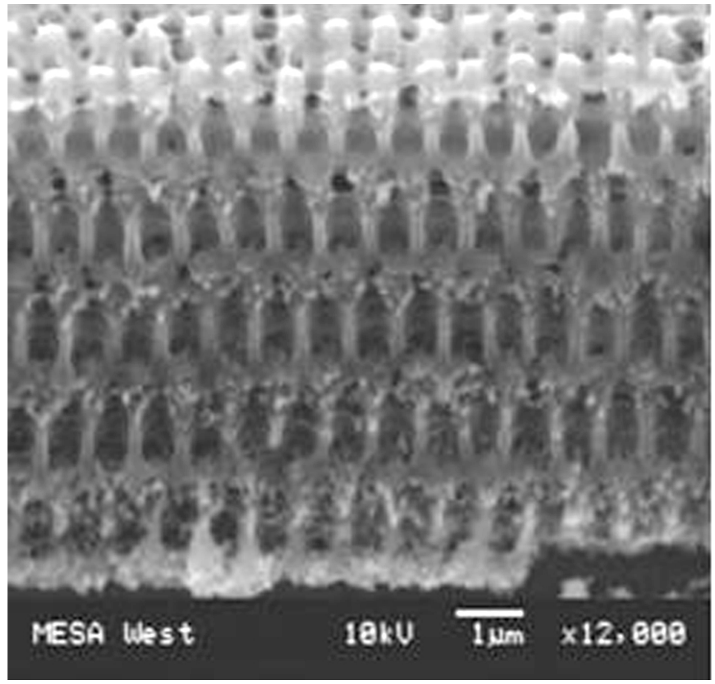
We are developing a revolutionary capability to fabricate large areas of nanostructures in a wide variety of materials. These nanostructured materials are important for many evolving scientific and national security applications, including: 1) nanoporous filter elements for high-surface-area sensors and chromatographic separators/mixers, 2) high-efficiency catalyst supports, 3) novel subwavelength optical filters, 4) ultrathin holographic correlators, and 5) nanostructured surfaces to control wetting phenomena and facilitate drug delivery. The capability to synthesize these new materials will ensure Sandia's continued leadership in the nanoscience field.

The goal of this work is to develop a fundamentally new method to fabricate large areas of nanostructured materials. We established collaborations with the University of Illinois at Urbana-Champaign (UIUC) and the University of New Mexico (UNM) and transferred the preliminary fabrication and modeling processes. Key objectives for FY 2007 include development of an optimized predictive design environment; design, fabrication, and characterization of a new mask for three-dimensional (3D) photonic crystals; and photopolymer chemistry, exposure, and development optimization.

We will extend our collaboration with Dr. J. Rogers at UIUC to pursue new fundamental mask design work and demonstrate novel phase imprint methods to fabricate nanostructures for 3D photonic crystals. We will also support a second collaboration with UNM to develop a predictive optical modeling package for these structures.

FY 2006 Accomplishments

We established close working relationships with UIUC and UNM, and transferred the preliminary fabrication and modeling processes. We correlated our mask diffraction and light propagation models (in GDcalc) with UIUC's code (GSolver). We also developed analysis codes to predict the reflection/transmission/absorption spectra of candidate 3D nanostructures, setting the stage for next year's predictive optimization code development.



Sandia made proximity nano printed SU-8 resist cross section

We successfully demonstrated the phase mask creation, photopolymer exposure/development, and atomic layer deposition (ALD) processes. We fabricated polydimethylsiloxane (PDMS) conformal phase masks using master molds transferred from UIUC. Photopolymer exposure/development was simultaneously demonstrated using the 10 conformal masks transferred from UIUC, and expanding the process to use a standard 6" wafer exposure tool to start along our area scale-up path. We began experimental and simulation investigations of optimal exposure wavelength, spectral bandwidth, intensity, and time, leading to successful 3D nanostructure fabrication in SU-8 photopolymer.

This process resulted in minimal shrinkage induced cracking across 4” coatings. To facilitate larger area fabrication, we explored three alternative photopolymer formulations with significantly reduced shrinkage characteristics, while maintaining good structural integrity for later ALD coating operations.

To pursue ALD process development in parallel with these other tasks, we coated more than 20 UIUC-fabricated 3D nanostructures with Al_2O_3 and ZnO, determining the appropriate ALD chemistries and temperature profiles to ensure uniform coating without structural collapse due to melting of the photopolymer. We developed a novel Al_2O_3 “skeleton” approach to enable deposition of high-temperature materials without structural degradation, and identified metal ALD processes for platinum and tungsten.

We performed near- to long-wave infrared spectroscopy to investigate promising structures for 3D photonic lattices, and our near-field scanning optical microscopy (NSOM) capability was expanded to accommodate through-sample illumination for phase mask characterization.

Significance

Important national security applications will benefit from the development of this nanostructure fabrication capability, including chemical-biological and nuclear nonproliferation sensing, photonic devices and architectures, adaptive and tunable electro-optical and radio frequency components, micropower, and novel energy generation. Key accomplishments include providing a method of large-area 3D structure fabrication using conventional lithography tools along with modeling to predict photonic band gaps. Dimensional stability of the structures was enhanced and demonstrated to 200 ºC.

We developed collaborations with UIUC and UNM to address research challenges relevant to DOE Office of Science strategies and Sandia’s nanoengineering and education objectives. Additional partnering relationships for technology development and eventual technology transfer are being developed.

Mid-Infrared Quantum Dot Emitters Utilizing Planar Photonic Crystal Technology

102615
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Project Purpose

The three-dimensional confinement inherent in quantum dots yields vastly different optical properties compared to one-dimensionally confined quantum well systems. Intersubband transitions in quantum dots can emit light normal to the growth surface, whereas transitions in quantum wells emit only parallel to the surface; a key difference to be exploited. This feature opens up a variety of quantum dot devices that have no quantum well analog.

Two significant problems limit the use of the beneficial features of quantum dots as mid-infrared (MIR) emitters. One is the lack of understanding concerning electrical injection of carriers into states that allow efficient optical transitions. Engineering of an injector stage leading into the dot can provide current injection into an upper dot state; however, to increase the likelihood of an optical transition, the lower dot states must be emptied faster than upper states are occupied. We are investigating engineering the confinement potential using our nanostructure growth experience coupled with model-based design to manipulate the tunneling rates from the conduction band energy levels.

The second issue is that InAs quantum dots have significant inhomogeneous broadening due to the random size distribution. While this may not be a problem in the long term, we can circumvent this issue by using planar photonic crystal (PC) technology that is available at Sandia to make devices with only a small number of quantum dots in the active region. Overcoming these challenges will require both creative device design and theoretical understanding of quantum dot intrasubband physics to realize the predicted advantages of quantum dot based emitters over quantum well devices in the mid-infrared portion of the spectrum.

Our work includes close collaboration with experts at the Mid-Infrared Technologies for Health and the Environment (MIRTHE) center at Princeton University, as well as theoretical photonic crystal modeling performed at the University of New Mexico (UNM).

FY 2006 Accomplishments

New Devices

We got off to a running start through the Sandia fabrication of grating couplers on Princeton-grown InAs self-assembled quantum dot (SAQD) material. The net benefit of the grating coupler was to improve the emission efficiency by two orders of magnitude over Princeton's previous devices.

Growth of InAs SAQD MIR material at Sandia

Generating material for this project was a responsibility shared between Princeton (molecular beam epitaxy [MBE] growth) and Sandia (metal-organic chemical vapor deposition [MOCVD] growth). These devices require control and growth of self-assembled InAs quantum dots on AlAs layers with AlGaAs capping layers. While this is a developed process for MBE, it has not been established for MOCVD.

We successfully resolved issues revolving around the growth of AlAs and AlGaAs at temperatures of 450 °C to 500 °C. After several experimental iterations, we achieved growth of InAs SAQDs on AlAs at Sandia. We will map out the experimental parameters further and then document these results for publication. The growth of these quantum dot heterostructures at Sandia is a key enabling result for this project.

Beyond growing InAs dots on AlAs for metrological purposes, we also designed and grew multilayer structures to investigate how SAQDs are capped.

These will not be functional devices; their purpose is to understand how SAQDs are capped in order to complement our wavefunction engineering effort. This material was prepared for transmission electron microscopy (TEM) and cross-sectional TEM analysis.

Photonic Crystal Processing

Photonic crystal devices consist of a hexagonal array of holes etched into a semiconductor substrate. The increased wavelength in the MIR allows optical lithography to be used to define the hole array. We designed PC cavity devices and developed processing techniques using material representative of PC cavity devices. The tolerances of the designed devices were tight and features as small as 1.6 μm had to be replicated accurately. We successfully demonstrated that this is possible with the current facilities.

Fabrication of these samples required a process that could etch holes in GaAs/AlGaAs structures several microns deep while maintaining straight sidewalls. Careful selection of the proper etch mask (SiO_2) and evaluation of the etch parameters enabled this result. Planning was facilitated by extensive literature searches combined with the expertise of Sandia's Compound Semiconductor Research Laboratory (CSRL) staff. This is the most difficult and critical part of processing for PC devices, so we are pleased that we were able to develop this capability in a short time period.

Theory

Our photonic crystal theory work is being performed in collaboration with UNM. The approach is to use finite difference time domain (FDTD) analysis to model PC structures and guide our design and fabrication efforts. The techniques will also be applied to understand emission from these devices after they are characterized. Sandia has a well established effort to model 3D photonic crystal structures. However, we did not have the capability to model 2D slab geometries needed for this project, so this capability was developed by a UNM graduate student and checked against literature to ensure accuracy.

Significance

The results of this LDRD project will enable the development of future InAs quantum dot based mid-infrared emitters. Our goal is to learn how much we can control and improve emission characteristics using photonic crystal cavities and surface plasmon couplers while at the same time improving the material through a combination of theory, growth, and experiment.

Development of SAQD emitters would enable novel optoelectronic devices with properties that are not presently available, such as a vertical cavity MIR laser. The development of MIR emitters for chemical and biological detection systems could provide technical superiority for defense and security systems.

Global Optimization for Nanomaterials

102642

J. C. Hamilton, J. Watson, W. E. Hart, E. Marquis, F. Leonard

Project Purpose

Calculating the energy of a given configuration of atoms is largely a solved problem. The inverse problem, "What configuration of atoms has the lowest energy?" is much harder. Major advances in nanoscience often occur when nature hands us an unexpected answer to this question, as, for example, the discovery of fullerenes. Humans, and especially computers, are not good at "thinking outside the box" and imagining something very different from what we already know.

These are issues of global optimization (GO), a notoriously difficult field. At Sandia and elsewhere, materials scientists presently perform GO by trial and error and/or simulated annealing. These methods often fail because they rely on random search and may revisit identical configurations millions of times yet miss the configuration with lowest energy. State-of-the-art GO algorithms are designed to remember and use search histories to minimize revisits to unproductive configurations.

We established a partnership between specialists in GO and materials scientists at Sandia and the University of California at Davis (UCD) to address two specific nanoengineering challenges: 1) alloy nanoclusters and 2) interface systems with variable interface densities.

Alloy nanoclusters serve as catalysts and as magnetic storage media. Experimentally, Sandia's new atom probe will reveal the shape, crystal structure, and distribution of different elements in alloy nanoclusters. GO will sort through the huge number of permutations of atomic species and the unusual crystal structures including, for example, five-fold symmetries, occurring in these clusters.

Interface systems with variable interface densities (e.g., surfaces, grain boundaries, and heterophase interfaces) occur in almost all applications of nanomaterials. Variable density requires that GO include adding and removing atoms, similar to the concept of the grand canonical ensemble of statistical mechanics. We will develop this capability. Our goal is to develop a tool combining human intuition with computer optimization to allow predictive engineering of nanomaterials.

FY 2006 Accomplishments

We formed a strategic partnership with UCD emphasizing global optimization and nanoengineering to study free nanoclusters and embedded nanoclusters.

Free nanoclusters

- We used temperature-accelerated molecular dynamics (TAD) to perform GO by simulated annealing at very low temperatures for very long times. We found the most stable cluster shape using this method. We also found that TAD is a superb tool for finding low-energy pathways for structural transformations of clusters and we discovered a cluster isomerization pathway lower in energy than any discovered previously during a decade of concerted effort by a number of investigators. Finding the pathway with the lowest activation energy is central to determining how stable a nanocluster will be in real-world applications.
- We applied tabu search, a state-of-the-art GO method making effective use of search history, to find the lowest energy state of a free 38 atom cluster. Tabu search was an order of magnitude faster than other GO techniques, in particular basin-hopping. We selected basin-hopping as a benchmark because it has often been used for optimization of cluster structure.
- We performed initial experimental work on Fe-Pt alloy nanoclusters. Atom probe examination requires sample fabrication as sharp tips. We prepared a set of tips by encapsulating Fe-Pt nanoclusters in a Cu matrix and then cutting tips using focused-ion milling. The tips were

then tested in the atom probe. For this first set of tips, we found that nanoparticle-matrix adhesion was not strong enough to allow atom probe examination.

Embedded nanoclusters

- Contrary to existing theories, we proved that edge energy is not responsible for size-dependent inclusion shapes in the Pb-Al alloy system. Strain energy was proven to be the key determinant of size-dependent shape effects and a new heuristic for assembling low-strain inclusions was developed.

Significance

Alloy nanoclusters are finding new uses in a variety of applications. For example, Au nanoclusters coated with Pd show great promise for the catalytic remediation of water contamination with trichloroethylene. Fe-Pt nanoclusters are promising for use in magnetic storage media. In both cases the arrangement of atoms in the cluster will be critical to the application. For Au-Pd catalysts, the dramatic catalytic activity is thought to be due to the arrangement of atoms at the surface and edges of the cluster. For Fe-Pt storage media, the important magnetic properties require a layer structure with Fe and Pt layers.

While these alloy nanoclusters are crucial for applications, understanding and modeling their behavior is difficult. The number of possible permutations of atom positions is large, making it difficult to search out the configurations with lowest energies that are most likely to be found experimentally.

These problems lie in the realm of global optimization. Simulated annealing is a well-known technique for finding minimum energy structures. Unfortunately, simulated annealing often requires extremely long simulations at very low temperatures to be successful. Such long simulations are generally impossible for problems of interest using conventional molecular dynamics or Monte Carlo methods. In order to dramatically extend annealing times at low temperatures we used new accelerated dynamics techniques, in particular temperature-accelerated dynamics.

We have shown that TAD is ideally suited to finding low-energy states. Furthermore, it is very good at finding the lowest-energy pathways between these low-energy states. This is important because these lowest-energy pathways determine reaction rates and thus the lifetime of the desired nanocluster configurations.

Laser-Based Microforming and Assembly

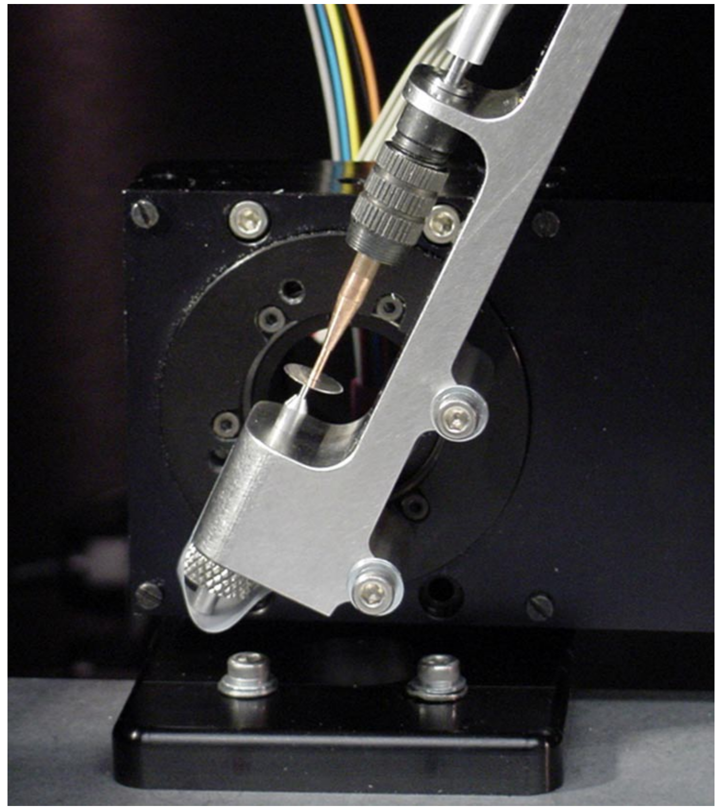
102659

J. A. Palmer, J. D. Arvizu, G. A. Knorovsky, D. O. MacCallum

Project Purpose

It has been shown that thermal energy imparted to a metallic substrate by laser heating induces a transient temperature gradient through the thickness of the sample. In favorable conditions of laser fluence and absorptivity, the resulting inhomogeneous thermal strain leads to a measurable permanent deflection.

The purpose of this project is to establish parameters for laser microforming of thin materials that are relevant to Microsystems and Engineering Sciences Applications (MESA)-generation weapon system components and confirmed methods for producing micrometer displacements with repeatable bend direction and magnitude. We realized precise microforming vectors through computational finite element analysis (FEA) of laser-induced transient heating that indicated the optimal combination of laser heat input relative to the material being heated and its thermal mass. We demonstrated precise laser microforming in two practical manufacturing operations of importance to the DOE complex: micrometer gap adjustments of precious metal alloy contacts and forming of mesoscale cones.



Mesoscale laser metal spinning apparatus

FY 2006 Accomplishments

Laser-based microforming of Neyoro-G™ (commercial gold-base alloy) strips and 304 stainless steel conical forms has been successfully analyzed with the coupled thermomechanical finite element code Calagio. FEA models consider plastic strains induced by heating by a laser, followed by conductive and convective cooling. We input critical material properties, such as the elastic modulus, as functions of temperature. The output established parameters for forming displacements with the desired 25-micrometer magnitude.

Preliminary results are encouraging. Ongoing efforts to tune the model parameters (such as the coefficient of convective heat transfer, mesh density, and simulation time step) have improved the run-time and accuracy of the results.

We analyzed laser microforming of a stationary (nonrotating) 304L metal disk, referred to as a cone preform, in a second FEA. The model considers the preform with a diameter of 6 mm and a thickness of 0.127 mm. A laser spot is scanned in a circumferential pattern across the top surface. We compared temperature distribution data from the FEA to thermocouple measurements from a validation experiment. Results indicate good agreement with the measured peak temperature.

Controlled laser microforming (bending) of Neyoro-G™ has been achieved for the first time in this project. A unique inductive sensor apparatus for microlaser forming of Neyoro-G™ strip was developed through a partnership with the Pennsylvania State University Electro Optics Center. Efforts are ongoing to configure the apparatus for the first known demonstration of laser microspinning of mesoscale conical forms. A literature search on the permanent material effects of laser microforming is also ongoing. This data is incorporated in finite element models.

Significance

Complex mechatronic products in the aerospace, automotive, and defense markets evolved to satisfy ever-changing demands for greater functionality, performance, safety, reliability, and economy. This trend fueled technical achievements in material and manufacturing systems, many of which are related to miniaturization. Beyond the domain of semiconductor microfabrication, the methods used to create and assemble miniature or mesoelectromechanical components with micrometer features and precise tolerances have also improved. Some traditional methods have been adapted to smaller scales. One example is thermal forming (so-called flame bending) of metals.

Before the 1990s, flame bending was applied more frequently in heavy industries, such as ship building and construction. Over the past decade, however, knowledge of flame bending has led to laser forming and, more recently, laser microforming of metals. Laser microforming has been successfully applied to create micrometer bends or adjustments in miniature metal components, such as reed switch contacts and disk drive read/write heads.

The significance of this research is that it advanced laser microforming of small, thin precious metal substrates and mesoscale conical forms. Laser microforming is the foundation for a disruptive mesomanufacturing system, including large deflection of metallic links, laser welding, and laser machining, that enables component assembly without fasteners or human intervention. This research supports the MESA vision by implementing science-based engineering in laser microforming research. Furthermore, it directly engages top-performing MESA Institute students in high-value research that will impact next-generation weapon system manufacturing initiatives.

Refereed Communications

D.O. MacCallum, G.A. Knorovsky, J.A. Palmer, J.D. Arvizu, "Precision Adjustment of Spring Contacts Using Laser Forming," in *Proceedings of the ICALEO 2006*, October 2006, CD-ROM.

Experimental and Theoretical Determination of Thermal Defect Generation on Silicon Surfaces for Control of Nanoscale Structures

102660
B. S. Swartzentruber, G. L. Kellogg

Project Purpose

The formation and stability of engineered structures at the micro- and nanoscale depends sensitively on a delicate balance between thermodynamics (the free energies of the configurations) and kinetics (the rates of various atomic processes). Our ability to guide the physical, chemical, electronic or magnetic properties of nanoengineered structures depends critically on a comprehensive understanding of the atomic-scale mechanisms that are active for particular material system and how these mechanisms influence the evolution of structures containing tens to tens of thousands of atoms.

In this project we will use a unique microscopic capability, a combined low energy electron microscope (LEEM) – scanning tunneling microscope (STM), to probe both the atomic-scale kinetics and macroscopic-scale thermodynamics involved in the earliest stages of thin-film growth on semiconductor surfaces. We will integrate dynamic microscopic measurements with state-of-the-art theoretical modeling methods to determine the dominant thermal defect present on Si(001) surfaces (adatoms, vacancies, addimers, advacancies) at elevated temperatures and to identify the physical or chemical modifications (mechanical stress, dopant species) that change their relative populations. The fundamental understanding derived from this project will be used to help direct and control the self-assembly of two- and three-dimensional (2D and 3D) nanostructures (e.g., quantum-dots) on semiconductor surfaces.

With all of the research that has been conducted to characterize the Si(001) surface, it is surprising that one of the most fundamental questions – what type of thermal defect populates the surface in equilibrium – remains unanswered. By quenching an engineered, ultraflat Si(001) terrace from elevated temperatures and observing the 2D islands that nucleate, we will determine if the majority species is adatoms or vacancies. First-principles calculations of formation energies and entropies will identify the exact type of defect (monomers, dimers, etc.) that is energetically favorable. The use of highly boron- and phosphorous-doped samples, which might change the charge state of defects, and the introduction of controlled impurities will be used to determine if it is possible to manipulate the defect populations by external means.

FY 2006 Accomplishments

We secured university and industry partners to leverage our efforts in this project. Prof. Franz. Himpsel of the University of Wisconsin-Madison, a world-renowned expert in the growth and characterization of surface nanowires, will join us to work on 1D and 2D self-assembled nanostructures in our bottom-up investigations of nanostructure formation with the LEEM-STM. Dr. James Hannon from IBM-Yorktown Heights has extensive expertise in the fabrication of free-standing nanowires. We hired Ezra Bussmann, the University of Utah, as a postdoctoral appointee to run the experiments on the LEEM/STM.

We performed detailed vibration measurements of the Elmitec LEEM system for comparison with our working atom-tracking STM. The Elmitec system had much higher vibrations at frequencies less than 60 Hz, which is not suitable for STM operation. After modifying the LEEM table configuration to accom-modate air isolation legs, we prototyped a system with borrowed legs. The improvement justified purchasing air legs for permanent setup. We acquired the legs and demonstrated a dramatic improvement in vibration characteristics. The LEEM performance was improved through isolation and repair of a small leak in the column. We are currently working to prepare a standard Si(111) sample for testing of atomic resolution of the STM with subsequent LEEM imaging.

Significance

Recently, a number of Sandia's mission-related technologies became invested in exploring the novel properties of nanostructures. These technologies include (but are not limited to) chemical sensors, water and gas purification, hydrogen storage, fuel cell applications, and the bioinorganic interface. The nanometer scale measurements enabled by our instrument will play a critical role in understanding and defining the function of nanostructures in these applications.

This work will generate the knowledge needed to engineer surface nanostructures in a reliable and controllable manner and, in so doing, will contribute to DOE and national initiatives on nanoscience and nanotechnology. By involving university faculty and graduate students, it will enhance the nation's educational competitiveness in science and technology.

Advanced Modeling and Simulation to Design and Manufacture High Performance and Reliability Advanced Microelectronics and Microsystems

102661
K. G. Ewsuk

Project Purpose

Within the legacy Sandia Engineering Analysis Code Access System (SEACAS) and the multiphysics SIERRA platform, Sandia developed and compiled an impressive science-based engineering capability to complete, among other things, stress modeling (JAS3D and Adagio), thermal modeling (Calore), process modeling (Skorohod Olevsky viscous sintering [SOVS] model and Riedel-Svoboda sintering model), and reliability modeling (FailProb).

However, despite this extensive capability, models like Adagio have been used primarily in a reactive or responsive mode to assess and troubleshoot specific problems that arise with materials and/or components in production. There is also considerable, but currently largely unrealized, value in using the science-based engineering capability within Sandia's computational modeling portfolio proactively to accelerate the integration of experimentally validated theoretical knowledge into design tools that engineers can use innovatively to realize products faster and cheaper, and with greater performance margins. Additionally, through a combination of characterization and modeling, more quantitative science-based understanding of the uncertainty in design margins can be established to improve confidence in the performance of specialty components.

We assembled an interdisciplinary team of scientists and engineers with expertise in materials processing and properties, materials characterization, and computational mechanics to integrate scientific understanding, experiment/testing, and predictive component and system engineering into a dramatically more responsive product realization process. Through a combination of characterization, modeling, and model validation, the team was tasked with drawing upon an appropriate combination of scientific discovery and engineering tool development to enhance the ability to apply Sandia's model-based engineering technology in product design and manufacturing. The focus of this work is on developing experimentally validated simulation tools that can be applied to complex microelectronics and microsystems to 1) improve designs for manufacturability, 2) better understand and control processing, and 3) reproducibly fabricate components with high performance and reliability. To complement our in-house expertise, we forged collaborations with experts in the field of advanced electronic materials in the Center for Dielectric Studies at the Pennsylvania State University and with experts in microstructure characterization at the University of Pittsburgh.

FY 2006 Accomplishments

We focused on defining and developing an enhanced science-based infrastructure to enable predictive compaction, sintering, stress, and thermomechanical modeling in "real systems," including: 1) determining materials properties and constitutive behavior for modeling; 2) developing new and improved models and modeling capabilities that are more representative of the physical phenomena being simulated; and 3) assessing existing modeling capabilities to identify the advances necessary to facilitate the practical application of predictive modeling technology. The more significant accomplishments are summarized below.

Advanced characterization equipment and techniques were developed and/or improved/enhanced to support parameter determination and testing and validation of predictive modeling.

- We designed a noncontact laser measurement system to characterize thick film sintering. The individual components of the system were designed and fabricated.

- The capability to complete controlled cyclic loading tests to characterize bulk materials properties/behavior was added to the thermal optical mechanical measuring instrument.
- We established the capability to characterize glass and composite viscosity, and we characterized National Institute of Standards and Testing glass viscosity standards.
- We developed a new technique to determine the stress-free temperature of a glass or composite to support more quantitative finite element (FE) stress modeling.
- Novel indentation and nanoindentation techniques were developed to characterize composite properties to support and validate FE modeling of composite structures.

To better help understand, predict, and control sintering behavior in real systems, we related mesostructure to observed and model-predicted continuum behavior in zirconia during sintering using a novel physical picture of nonideal microstructure evolution obtained using quantitative microstructure analysis.

We established enhanced modeling capabilities that are being tested/validated.

- Models of a two-phase (composite) were developed and applied to demonstrate the feasibility of predicting composite properties from the properties of the constituents of the composite.
- We used Calagio to complete coupled thermomechanical modeling of a multilayer microelectronics package heated with an embedded resistor. Model predictions compared favorably to experimental measurements.
- The Sandia GeoModel was identified as an improved model to describe the constitutive behavior of ceramic powders for compaction modeling and the GeoModel parameters were determined for three ceramic powders.
- We fabricated and characterized zinc oxide samples in hydrostatic and triaxial compression to obtain the properties and constitutive behavior necessary to model zinc oxide compaction and to enable coupled compaction and sintering modeling of zinc oxide.
- The Riedel-Svoboda and SOVS models within JAS3D were updated to the most recent version of JAS3D to enable coupled compaction and sintering modeling.

We assessed phenomenological, constitutive, and continuum models for sintering to provide guidance on how to refine and improve existing models and modeling capabilities to enhance Sandia's modeling portfolio. Subincrementation and the use of sheet elements were identified as means to improve the efficiency of modeling sintering of multilayer microelectronic structures.

Significance

This project resulted in: 1) the development of new characterization equipment and capabilities to determine materials properties and constitutive behavior critical to predictive modeling; 2) new, improved, and up-to-date models and modeling capabilities that are more representative of the physical phenomena being simulated; and 3) the identification of advances necessary to facilitate the practical application of Sandia's predictive modeling technology.

Representative materials properties and behavior are absolutely critical for predictive modeling. The two new characterization capabilities developed will afford accurate in situ measurement of sintering shrinkage and in-plane sintering stress of thin film, thick film, and bulk materials to support predictive sintering modeling of multimaterial multilayer microelectronics. Among other things, these new characterization capabilities have established the ability and flexibility to more efficiently complete controlled experiments that, with DAKOTA, will enable the more efficient determination of the parameters needed for predictive sintering modeling. Additionally, we developed a powerful new technique to determine the stress-free temperature of a glass that will enable more accurate finite element modeling of residual stresses in glass seals, and in turn, more predictive reliability modeling.

The work completed to relate green density, densification, and microstructure evolution provides the physical picture of microstructure evolution that must be considered along with observed and simulated continuum behavior to better understand, predict, and control sintering behavior in “real,” nonideal systems. This has provided valuable new insight into how the heterogeneities in real systems can contribute to sintering damage (e.g., cracks) that can affect performance and reliability.

We now have a firm foundation to start addressing the effects of heterogeneous and constrained sintering in more representative sintering models. Additionally, the capability to quantitatively characterize and describe heterogeneous microstructures can be applied to better understand, control, and optimize nonideal sintering in “real” materials, which will be essential for the controlled processing of nanomaterials and microsystems.

The modeling work completed to demonstrate the feasibility of predicting composite properties from the properties of its constituents validates the use of a micromechanics approach to understand microscale behavior and how it relates to the macroscale. In turn, this also represents an important and critical step to predicting, understanding, and controlling nanoscale behavior. Furthermore, we also demonstrated the value of coupled modeling to better understand and control complex interrelationships in more complex systems.

Our assessment of phenomenological, constitutive, and continuum models for sintering provided valuable guidance on how to refine and improve existing models and modeling capabilities, as well as to further enhance Sandia’s modeling portfolio. We have identified a clear need to better understand the effects of materials and process nonidealities on the sensitivity and uncertainty of simulation results, (e.g., versus the ideal conditions currently assumed). Additionally, improved models that provide the potential for more detailed understanding relative to the physical micro/macrostructure and that enable greater scientific understanding were identified. Finally, we also identified enhancements to improve modeling efficiency.

RF/Microwave Properties and Applications of Directly Assembled Nanotubes and Nanowires

102662

M. Lee, A. A. Talin, C. Highstrete

Project Purpose

It has been speculated that single-walled carbon nanotubes (SWCNTs) and semiconductor nanowires (SNWs) may have unique and desirable high-frequency characteristics that can lead to potentially disruptive advancements in radio frequency (RF)-to-microwave technologies for very-high-speed electronics and chemical/biological sensing. Despite extensive efforts to characterize and exploit DC electrical properties of such nanomaterials, even the most fundamental RF and microwave properties of CNTs and SNWs remain nearly unexplored and certainly undeveloped.

One of the major upcoming frontiers in nanomaterial research will be in high-frequency properties and applications. To open this frontier will require a focused effort to measure and model accurately the basic electrodynamic response of CNTs and SNWs across the range of frequencies relevant to high-speed communication, computation, radar, etc. (i.e., MHz up to tens of GHz). Once the electrodynamic physics is better understood, a number of innovative applications can be envisioned. For example, pure nanowires and nanotubes are believed to exhibit ballistic charge transport, a phenomenon that does not exist in micro- or macroscopic materials. This novel transport mechanism could be exploited to construct a new class of extremely power-efficient, frequency-agile RF and microwave oscillators and detectors.

SWCNTs and SNWs can also be chemically functionalized with specific receptors. These nanomaterials could lead to the development of AC nanodevice components that are chemically tunable and thus usable in resonant chemical and biological agent sensors with sensitivity several orders-of-magnitude better than existing DC electronic methods.

FY 2006 Accomplishments

We initiated collaborations with two university research groups to provide new expert-level research training opportunities in nanotechnology. The partnerships are with Prof. David Dunlap at the University of New Mexico (UNM) and Prof. Theresa Mayer at Pennsylvania State University, whose research groups bring necessary and integral resources and experience to this project.

We built an AC dielectrophoresis (ACDEP)-directed assembly at Sandia to assemble arrays of single-walled carbon nanotubes (SWCNTs) and silicon nanowires (SiNWs) onto broadband coplanar waveguides (CPW) in a way that aligns the nanotubes and wires with the electric field polarization of the propagating RF and microwave fields in the CPW, thus maximizing the interaction between the electromagnetic field and the nanomaterial.

We exploited a deep reservoir of experience with ACDEP at both Penn State and Sandia to get the new ACDEP capability up and running very rapidly. Purified SWCNT material, with and without DNA surfactant, was prepared in suspensions suitable for ACDEP assembly at Sandia. SiNWs in a wide range of dopant concentrations, ranging from n+ to undoped to p+, were grown and characterized at Penn State and prepared into suspensions suitable for ACDEP assembly.

We showed the ability to measure frequency dependent reflection and transmission coefficients that result unambiguously from SWCNT and SiNW arrays on CPWs. By virtue of their very small size, SWCNTs and SiNWs have very small interaction cross-section with the electromagnetic field. To have confidence that the signals measured are due to the

SWCNTs or SiNWs and not artifacts, a very high level of systematic measurement reproducibility is required to eliminate measurement-to-measurement variations. We achieved extremely high systematic reproducibility and signal-to-noise between 10 to 100 times better than what other research groups have reported. We developed the ability to fabricate CPWs with extremely reproducible microwave characteristics, which allows extraction of nanomaterial response with very high confidence.

Significance

The coplanar waveguide (CPW) is the basic broadband RF-to-microwave electrode geometry that guides high-frequency signals to microwave device components on a chip. To adapt nanomaterials into a high-frequency circuit, it is necessary to be able to assemble single-walled carbon nanotubes (SWCNTs) and silicon nanowires (SiNWs) onto CPWs in such a way as to be simultaneously compatible with both the 50 ohm impedance necessary for broadband microwave signals and components and the small dimensions required for high-purity SWCNTs and SiNWs. The integration of microwave and nanomaterials has not been accomplished in a reproducible way before. Our results using ACDEP essentially solve this integration problem in a compact, reproducible, and cost-effective manner.

The broadband RF-to-microwave reflection and transmission measurements carried out on SWCNTs and SiNWs mark the first successful, quantitative measurement of the basic high-frequency properties of such nanomaterials. Previous efforts have not had sufficient signal-to-noise or systematic reproducibility to give reliable or accurate results.

Two graduate students are using this project to pursue their Ph.D. thesis research in nanotechnology, using resources both at Sandia and at their home academic institutions. Training opportunities such as this in a rapidly developing area of nanotechnology will contribute to continued US leadership in nanoscience and nanoengineering for the next generation.

Refereed Communications

C. Highstrete, E.A. Shaner, M. Lee, F.E. Jones, M. Dentinger, and A.A. Talin, "Microwave Dissipation in Arrays of Single-Wall Carbon Nanotubes," to be published in *Applied Physics Letters*.

Quantum Dot Logic to Extend Moore's Law

102663

E. P. DeBenedictis, M. Ottavi, S. Murphy, A. A. Prager, J. A. Floro

Project Purpose

The purpose of the project is to understand a new physical device that is a candidate to replace transistors in the development of computers and associated theory.

The computer industry has grown for many decades driven by advances in transistor performance. There is broad acceptance that industry's pipeline of advances becomes empty at some point in the future. This project seeks to understand a proposed device called a quantum dot cellular automata (QDCA). Understanding this device requires more reliable fabrication/manufacturing technology, improved understanding of how to incorporate it into circuits, and an assessment of the impact on computers and their ability to solve important problems.

FY 2006 Accomplishments

We fabricated several samples of "quantum fortress"-style QDCA cells and are in the process of examining them experimentally.

We developed a guide for applying QDCA devices to the problem of computer design. This guide outlines general principles for combining QDCA devices into logic circuits, providing a method for estimating and controlling complexity, power dissipation, and other factors. This guide will also influence future device designers by giving them an understanding of which device characteristics are important, thus permitting them to develop devices that are better suited to computer design.

Significance

The project demonstrated the value of combining physical and computer science.

Sandia has made substantial investments in infrastructure for creating leading-edge nanodevices at the Center for Integrated Nanotechnologies (CINT) and the Microsystems and Engineering Sciences Applications (MESA) facilities. Sandia is also at the frontier of computing with groups that develop the most powerful computers of the day and program them to solve problems important to national defense and science.

The vision for combining these two capabilities has Sandia on the forefront of discovery and well-positioned to invent and reduce to practice devices that will power computers over the next several decades, including understanding how to organize the devices to be effective for computing.

This project involved senior Sandia technical staff in both physical and computer sciences. These Sandians worked with university researchers at Notre Dame and the University of Virginia in a collaborative arrangement, importing new external knowledge into the Sandia environment.

Other Communications

M. Ottavi, S.E. Frost-Murphy, E. DeBenedictis, M. Frank, and P. Kogge, "Design and Characterization of a Clock Distribution Circuit for QCA," submitted to *IEEE Transactions on Nanotechnology*.

S.E. Frost-Murphy, M. Ottavi, M.P. Frank, and E.P. DeBenedictis, "On the Design of Reversible QDCA Systems," Sandia Report SAND2006-5990, Albuquerque, NM, 2006.

M. Ottavi, S.E. Frost-Murphy, E. DeBenedictis, and P. Kogge, "Partially Reversible Pipelined QCA Circuits: Combining Power and Throughput," submitted to *IEEE Transactions on Circuits and Systems*.

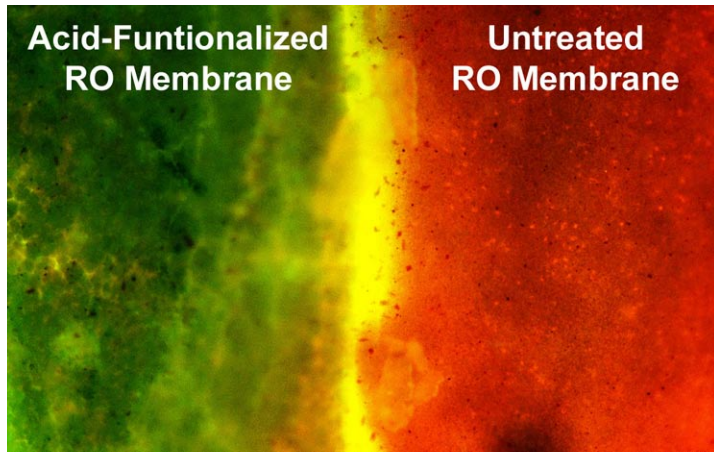
Creation of Water-Treatment Membrane Technologies with Reduced Biofouling

102737

S. J. Altman, H. D. Jones, C. K. Ho, C. J. Cornelius, P. G. Clem, A. Y. Hsu, W. E. Hart

Project Purpose

The overall goal of this project is to use high-performance computing to direct polymer, material, and biological research to create the next generation of water-treatment membranes. We made both physical and chemical modifications to membranes to increase resistance to biofouling. This is done by 1) integrating in situ chaotic micromixers onto the membrane surface to maximize mixing and 2) utilizing acid-functionalized polyphenylenes and polysulfones to improve chlorine and biofouling resistance. Modeling is used to optimize micromixer placement and membrane design. The three components of the project are: 1) creation of novel membranes; 2) computational optimization of membrane design; and 3) membrane testing.



Epifluorescent image showing the boundary on a RO membrane surface where the left-hand portion has an acid-functionalized coating showing a much greater number of micro-organisms on the noncoated portion of the membrane.

FY 2006 Accomplishments

We developed two types of novel membranes this year. We used MESA (Microsystems and Engineering Sciences Applications) direct-writing capabilities to print ultraviolet (UV)-curable micromixers on membrane surfaces to maximize turbulence and minimize stagnant regions on reverse osmosis (RO) membranes. Avoiding surface stagnation minimizes the deposition of micro-organisms on the membrane surface, retards the biofouling process, and prolongs useful membrane life. Various iterations of line and chevron heights, spacings, and "phase angle" were designed and printed to increase turbulence on the membrane surface. The second approach uses acid-functionalized polyphenylenes to make chlorine and biofouling resistant membranes. Six different polymers were synthesized and one tested on a membrane surface.

We created a three-dimensional (3D) computational fluid dynamics (CFD) model of four of the micromixer designs, as well as a membrane without micromixers. Numerical results show that the inclusion of micromixers can increase mixing and "scouring" along the membrane surface by a factor of three or better. Vortical, spiraling particle paths are induced by chevron-shaped features that enhance the mixing behavior. In addition, we are making progress in linking the 3D CFD model with the optimization DAKOTA toolkit. We developed a 2D CFD model in GOMA linked to DAKOTA. Thus far, the objective function decreases with decreased coverage and increased diameter, which points to a design that favors smaller mixers in conjunction with large area coverage.

Two crossflow membrane testing devices are being used for membrane testing. These systems monitor pressure, flow rate, temperature, and permeate flux in real time. We tested a total of eight membranes. Results show promise for the acid-functionalize coated membranes. Epifluorescent imaging gives a strong visual indication that there are more micro-organisms growing on the uncoated portions of the membrane than the coated portions. Testing of a chevron-embossed RO membrane also shows promising results. Areas with fewer microorganisms can be seen by both visual inspection and hyperspectral imaging of the membrane. In addition, the performance of the membrane also seems to have improved, as we observed an increase in flux with time for the embossed membrane.

The experiments also help validate the modeling results. We observed a qualitative correlation between regions of modeled high shear-stress and experimental low microbial growth (and vice versa). The experimental result that higher permeate flux occurred in the membrane with chevrons is supported by simulations that showed the overall mass transfer (scouring) along the surface of the membrane was three times greater with the chevrons than without.

We performed in situ monitoring of biofilm fouling using both Fourier transform infrared (FTIR) spectroscopy and wavelength-dependent UV-photoluminescence. In FTIR, bacteria display distinct protein amide group infrared absorption spectra that differ for each specific compound. This "fingerprint" provides a sensitive metric for biofilm formation. Biofilms of *Pseudomonas fluorescens* were successfully imaged by both microscope-based FTIR (top view) and attenuated total reflection (ATR) (signal bounced off an ATR crystal/biofilm interface) measurements.

Significance

Solutions are needed to produce fresh water economically as populations and agricultural demands continue to grow. If future water needs are not addressed with improved desalination and water remediation technologies, it is anticipated that water shortages could escalate globally. Membrane-based separation processes, such as reverse osmosis (RO) and ultra- or nanofiltration, are commonly used in industrial applications such as desalination, waste-water treatment, and power generation. The major problems associated with membrane-based separation processes include fouling and high pressure loss, which decrease the efficiency of the filtration while increasing operation costs.

If our long-term goals are successful, we will have developed an innovative water filtration technology with an integrated science-engineering, experimental-numerical based approach. Work conducted this year lays the groundwork for enhancing water-filtration membranes such that their filtration capabilities increase. We assembled an interdisciplinary team exploring engineering-sciences as well as optimization of design through high-performance computing. Through our novel RO membranes and examination of the biofouling on the membranes, scientific discovery will also be possible.

This work supports the goals and activities of Sandia strategic management units including Energy, Resources, and Nonproliferation; Science, Technology, and Engineering; and Homeland Security and Defense. Our use of science-based engineering to understand the physical phenomena behind how membranes work and to design novel membranes, coupled with the use of numerical simulation that captures the governing physics to drive the membrane design aligns this project well with Sandia's science-based engineering for transformation (SBET) strategies. Model validation and system testing also tie this project with SBET.

The results of this work will ultimately impact the nation's ability to provide purified water for industries ranging from water treatment to power plants. Competition for decreasing supplies of water could lead to a fundamental source of conflict. Thus, technologies that reduce this conflict, through increasing water supply, will enhance US national security.

The Nanoscience, Engineering, and Computation Institute at Sandia (NECIS)

103006

J. L. Lee, S. S. Collis, J. A. Zimmerman

Project Purpose

The purpose of the Nanoscience, Engineering, and Computation Institute at Sandia (NECIS) project is to conduct research activities that integrate nanoscale physical and biological science with computational science, and to improve the nation's competitiveness in nanotechnology by building strong research collaborations between Sandia and academia. NECIS provides Sandia with research experience at the intersection of experiments and computational science for nanotechnology. NECIS also creates a pipeline of future Sandia employees who possess critical skills in the key technical areas of nanotechnology and advanced computing. NECIS research activities can inspire and expedite multidisciplinary breakthroughs in nanotechnology that will provide the foundation for Sandia's response to the American Competitiveness Initiative. Important areas of concern are training our nation's future workforce that will drive US technical innovation and leadership; and encouraging creative, leveraged, and seamless research collaborations between national laboratories, academia, and industry.

FY 2006 Accomplishments

NECIS Summer Research:

The summer collaborative research effort included 48 student interns from 35 universities working with a total of 54 Sandia staff. Targeted areas that support DOE strategies in energy, science, and defense were defined for the summer research activities:

- Nanoscale defects in materials
- Nano- and microfluidics
- The nano-to-micro interface
- Nano/bio synergy
- Enabling computational nanoscience.

Results from these summer research projects were documented in extended abstracts and collectively will be published as a SAND report.

NECIS Short Courses and Seminars

We held two short courses (Extended FEM for Multiscale Systems and Mortar Methods for Mesh Tying) and two seminar series to highlight the multidisciplinary nature of nanoengineering and to engage with academic partners in nanoengineering research. The first "special" seminar series featured university faculty speakers working at the intersection of experimental nanoscience, modeling, simulation, and computer science. The second "student" seminar series featured Sandians presenting talks showcasing research where nanoengineering impacts Sandia programs. Both seminar series were well-attended, with several talks drawing standing-room-only audiences.

NECIS Workshop

A NECIS workshop on "Multidisciplinary Approaches to Science for Nanoscale Interfaces" assembled top researchers from academia, national laboratories, and industry to discuss and disseminate important ideas and issues on this topic. Workshop discussion topics included issues that are germane to doing leading-edge research, including identifying challenges that prevent researchers from doing predictive science (in this case, on nanoscale interfaces), proposing new ways for researchers with varied expertise and backgrounds to collaborate to stimulate innovative research, and preparing the next generation of scientists to do research on interfaces.

We summarized the workshop's technical presentations, discussion of key issues, and recommendations for paths forward. It is the intent of this document to foster further discussion in the research community on how to enable cross-sector, multidisciplinary collaboration in cutting-edge research areas, how to sustain US technical leadership, and how to identify and prepare this country's next generation of technical leaders.

In addition, we set up a NECIS Web site as a mechanism for information sharing among NECIS participants that promotes opportunities for the cross-disciplinary interactions that often underpin innovation.

Significance

The research performed by NECIS collaborations between Sandia staff and university faculty and students is expected to yield publications in high-impact, peer-reviewed journals.

Many NECIS projects produced results that will help Sandia select and build new research areas. While each NECIS collaborative project is a standalone research effort, they are in many cases inspired by current projects or lay the foundation for future projects. For example, NECIS projects in nanofluidic devices relate to Sandia's homeland security mission and bioagent detection efforts. Several NECIS projects model, simulate, and characterize the behavior of nanoscale defects in materials that can help predict the performance of materials in critical applications such as nuclear weapons. NECIS projects at the intersection of biology and nanoscience may impact applications such as biofuel process development and water quality in the Energy, Resources, and Nonproliferation strategic management unit.

Nanotechnology and advanced computing are critical to the national security mission, and the current shortage of U.S. citizen engineering and science students with appropriate skills makes it difficult to fill permanent positions in these fields at Sandia and other national laboratories. NECIS enabled state-of-the-art research and research training that will help Sandia and the nation develop critical research skills in these fields through academic collaborations with top universities.

Other Communications

S.S. Collis, J.L. Lee, and J.A. Zimmerman, "NECIS Summer Proceedings 2006," Sandia Report SAND 2006-6564P, Albuquerque, NM, 2006.

Ultrahigh Mobility 2D Electron Systems for Science and Technology

103926
J. L. Reno, M. P. Lilly, W. Pan

Project Purpose

The purpose of this project is to develop a full science-based conceptual design for a "100 M" mobility molecular beam epitaxy (MBE) machine to meet the clear and compelling needs of the condensed matter physics community. This revolutionary new MBE growth reactor will be designed to produce two-dimensional (2D) electron systems having electron mobilities in excess of 100 million cm^2/Vs, or roughly three times the existing world record. We will outline the conceptual design in a full report documenting the design elements and containing a roadmap to the construction, installation, commissioning, testing, and initial growth procedures. The roadmap will represent a distilled snapshot of the best of the community's collective knowledge of ultrahigh mobility growth techniques, instrumentation, and procedures. The guiding principle in our design is that ultrahigh mobility (i.e., background pressure and impurity levels) takes priority over other common considerations such as flexibility. Due to this guiding principle, the typical trade-offs in designing a modern MBE system will not be necessary.

FY 2006 Accomplishments

We developed the conceptual design in consultation with other growers of high mobility material, MBE equipment manufacturers, and users of high mobility material. A list of three main issues that need to be addressed was determined by the growers, users, and suppliers based on what are believed to be the present limiting factors. These three issues are: improving residual background doping/material purity, assuring long periods of continuous operation, and material uniformity. We then looked at what are believed to be the sources of contamination and factors limiting continuous operation. Starting with this list of objectives, we consulted with an MBE equipment supplier to compose the system design.

We started with a basic existing system and determined the possible needed modifications. We evaluated the modifications based on their feasibility, projected cost, and functionality. For example, using the supplier's computer models, we determined what types of effusion cells should be used, their distance from the substrate and their angles. We also considered some possible advanced components that would need to be developed.

Actual production of extremely high mobility materials takes more than just the MBE system. It requires material growth, evaluation, and feedback. Consulting with both experimentalists and theoreticians involved in the area, a plan was developed for initial qualification and growth optimization of the system.

We completed the final report outlining the design of the 100 M mobility machine, with justifications for each design element. The report serves as a complete roadmap for realizing a new high-mobility growth capability to enable new condensed matter physics discoveries for the next decade and beyond.

Significance

For almost three decades, low-dimensional (i.e., 0D, 1D, and 2D) electron physics has been one of the most active, productive, and exciting fields in science. The field of quantum Hall physics alone (a subfield of low-dimensional electron physics) has attracted mathematicians, high energy theorists, quantum computationalists, and a large community of condensed matter theorists and experimentalists, and has seen two Nobel prizes. Advances in this field have revolutionized our thinking about possible states of matter and emergent collective phenomena, and new experimental discoveries continue to occur at a rapid rate. Other subfields in low-dimensional physics include ballistic transport, quantized conductance states in 1D, single electron transistors, the metal-insulator transition, and Luttinger-liquid and other nonFermi liquid electron states.

What has enabled these revolutionary discoveries in fundamental condensed matter physics has been continuous advancement in the state of the art of ultrapure semiconductor materials. The standard quantitative measure of material quality for this purpose is electron mobility. Measured in units of square centimeters per volt-second (cm^2/Vs), electron mobility describes the drift velocity of an electron in response to an applied electric field within the material.

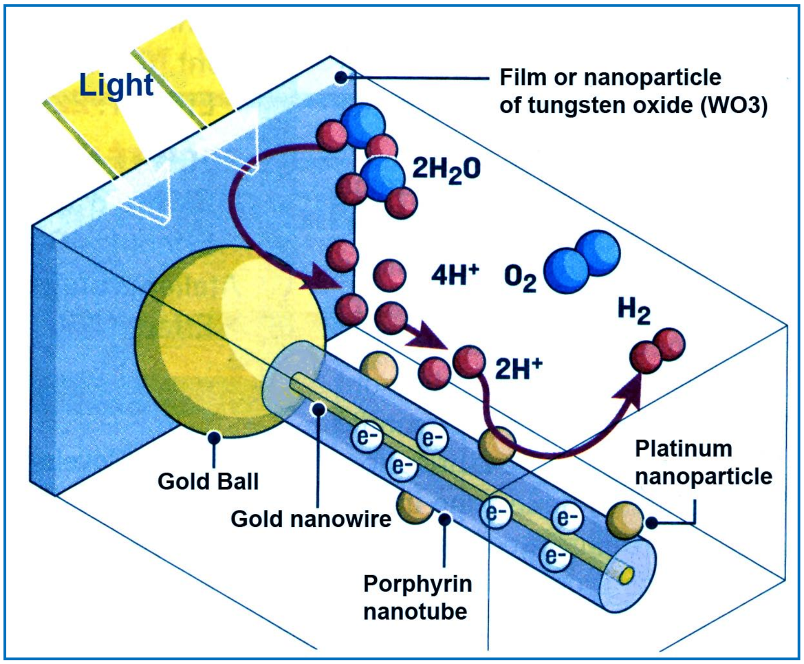
Large values of mobility are equivalent to large electron mean free paths, long inelastic scattering lengths, and fast electron transit times. Large mobility values are also equivalent to a low disorder potential, meaning that the delicate collective electron states, arising due to electron-electron Coulomb interactions, are not destroyed by disorder. High mobility allows stringent tests of the fundamental physical limits on high-frequency transistor/detector speed, bandwidth, and sensitivity. Finally, large mobility translates to intrinsically narrow electron energy level widths and long electron coherence times, enabling spectroscopy of quantum dot energy levels and the possibility of solid-state qubit formation for quantum computing applications. For the past quarter century, steady advances in the MBE growth of high-mobility AlGaAs/GaAs 2D electron systems have led to new fundamental discoveries in condensed matter physics.

Mission Technologies

Mission Technologies investments aim to create or accelerate the technical innovations that provide solutions to difficult national security problems. They also seek to extend the boundaries of the science and technology into fertile new areas that will support current and future missions. Mission Technologies is composed of four investment areas: Energy and Infrastructure Assurance, Military Technologies and Applications, Nonproliferation and Assessments, and Homeland Security.

Energy and Infrastructure Assurance projects support Sandia's national security mission by developing and applying technologies in three primary areas: energy surety, global nuclear future, and water. While work across these areas is primarily focused on technical challenges within the US, the challenges reflect a fundamentally global dimension as well.

Projects focus on new concepts, ideas, and technologies needed to increase the sustainability (availability and utilization) of domestic water and energy supplies, improve the security and reliability of our energy infrastructures, reduce our dependence on imported energy, and minimize the impact of energy production and utilization on the environment and natural resources.



Project 93563, Water-Splitting Nanodevices for Solar Hydrogen Production

A prototype water-splitting nanodevice based on the porphyrin nanotubes, which may function as one of the two coupled photocatalysts required to split water into hydrogen and oxygen. In the nanodevice shown, tungsten oxide photocatalytically evolves oxygen using UV and blue light, while producing electrons that move through the gold conductor to the nanotube. There, the electrons are promoted by the photoactive porphyrin nanotubes to high enough energy to reduce water to hydrogen using visible light.

Project 93563, Water-Splitting Nanodevices for Solar Hydrogen Production, for example, is making fundamental science breakthroughs that could enhance solar hydrogen cell efficiency; spawn new types of nanoelectronics, nanophotonics, and small power sources; benefit energy, weapons, and nonprolif-eration missions; and help establish a hydrogen-based economy.

The project team is making sub-stantial progress in fabricating and testing nanodevices. They demon-strated two viable methods for using porphyrin nanostructures in water-splitting nanodevices. Their initial results demonstrated hydrogen evolution from porphyrin nanotubes with adsorbed platinum nanoparticles when irradiated with visible light. They are combining experiment and computer simu-lations to develop a mature understanding of key molecular interactions and electron- and energy-transport mechanisms.

Homeland Security investments are a key component in enabling Sandia to meet its mission in defending the homeland against terrorist attack. Research activities pursue science and technology advancements in information analysis and infrastructure protection, borders and transportation security, and emergency preparedness and response.

The goal of the Homeland Security investment area is to provide technologies, systems, and solutions that allow the US to anticipate and prevent the use of weapons of mass destruction, as well as the capability to respond and recover if terrorist events do occur.

Military Technologies and Applications investments are about strengthening national security by anticipating and delivering high-impact solutions to a broad range of defense challenges.

This mission is accomplished in large part by applying Sandia's engineering, science, and technology base to systems-level problems in command, control communication, intelligence, surveillance and reconnaissance; energetics and homeland defense/force protection; integrated military systems; science and technology products; and transformational analysis and systems.

For example, Project 67035, Novel Processing, Affordable Motion Compensation, and Mode Multiplexing for Miniaturized Synthetic Aperture Radar (SAR), is working to create smaller, less expensive, higher-performance system architectures that could lead to current and future miniaturized SAR systems having unprecedented flexibility.

The team enhanced the autofocusing algorithm to allow the final image processing to compensate for motion measurement errors more severe than previously thought possible. Other project achievements are expected to result in significant reduction in size and cost of the inertial measurement unit (IMU), a key goal in reducing overall system size and cost. This project is evolving high performance radar from an exotic sensor on limited aerial platforms to a ubiquitous tool available to the ever-growing fleets of small unmanned aerial vehicles.

Project 67035, Novel Processing, Affordable Motion Compensation, and Mode Multiplexing for Miniaturized Synthetic Aperture Radar (SAR)

Real-time SAR image of golf course (4 inch resolution at 4 km range) from Sandia's miniSAR radar, using novel, LDRD-developed software architecture.

Reducing the nation's vulnerability to threats from the use of weapons of mass destruction and weapons of mass effect is the primary goal of the Nonproliferation and Assessments investment area. This includes destruction from nuclear, biological, or chemical weapons, and effect from cyber, radiological, or explosive attack.

Investments promote a set of capabilities and products that will enable the continued development of organized, system-level responses to national security challenges. Projects strive to fill knowledge gaps and to strengthen capabilities for detection, awareness, and international security.

Energy and Infrastructure Assurance

Fully Integrated System Dynamics Toolbox for Water Resources Planning

67052

V. C. Tidwell, H. D. Passell, J. L. Krumhansl, L. A. Malczynski

Project Purpose

Public mediated resource planning is quickly becoming the norm rather than the exception. Unfortunately, supporting tools are lacking that interactively engage the public in the decision-making process and integrate over the myriad values that influence water policy. To support Sandia's efforts to develop such a tool, we propose to create a modular and generic resource planning "toolbox."

The technical challenge lies in the integration of the disparate systems of hydrology, ecology, climate, demographics, economics, policy, and law, each of which influences the supply and demand for water. Specifically, these systems, their associated processes, and most importantly, the constitutive relations that link them must be identified, abstracted, and quantified. In this way, the toolbox will form a collection of process modules and constitutive relations that the analyst can "swap" in and out to model the physical systems important to their problem. This toolbox, with all of its modules, will be developed within the common computational platform of system dynamics linked to a geographic information system (GIS), allowing analysis of spatially distributed data.

This resource planning toolbox will provide Sandia with a unique tool for assisting local, national, and international water managers in resource planning. Additionally, development of this toolbox and interactive modeling environment will position Sandia as a clear leader in integrated modeling of water resource systems and will provide the technical foundation and framework for creating an interactive, icon-driven system dynamics modeling application for commercial distribution, and thus represents an important intellectual property development for Sandia.

FY 2006 Accomplishments

In this project's final year, we continued to develop key physical and economic process modules and integrate them into the toolbox. We coded new river-routing schemes and developed operational rules for the Middle Rio Grande's six reservoirs. We integrated the Española and Socorro Basin groundwater models into the Rio Grande model, thus providing a broad basin model stretching from the Colorado border to Caballo Reservoir.

In order to estimate watershed runoff, soil moisture storage, and groundwater recharge subject to various precipitation events, we completed an operational land surface module. We developed supporting GIS applications for preprocessing of data for the land surface module; specifically, precipitation, temperature, soil type, vegetative cover, and basin physical characteristics. Accompanying the physical hydrology and land surface modules is a set of routines for modeling water quality that addresses the aqueous chemistry of conservative solutes.

The modules consider mixing and dispersion processes between the river and the conveyance system, as well as the river and the shallow aquifer. We also integrated point sources, including urban storm and wastewater discharges and nonpoint sources from irrigated agriculture, into the water quality module. The new fish mortality module now uses the water supply and water quality outputs of the model. The module is generic in design but parameterized for the characteristics of the Rio Grande silvery minnow. The model relates changes in river characteristics to the evolving life stages of the minnow and allows users to investigate the impact of fish migration (e.g., diversion structures) and off-stream refugia on population vitality.

This year we greatly expanded the economic and demographic sectors of the model, allowing us to explore how different regional growth schemes influence economic development, population growth, and ultimately water supply. By growth schemes, we mean the ability to test different development pathways following growth in a particular industry (i.e., tourism, chip manufacturing, agriculture). As the industry grows, it generates jobs that encourage migration and growth in the commercial and residential sectors. From this information we can calculate increased demands on surface/groundwater supply, environmental impacts, mix and type of jobs, and economic benefits.

We also developed a water banking module that allows water trading among different stakeholders (irrigators, American Indians, municipalities, industry, and the environment). The nature of the trades and the resulting water allocation structures are investigated under an oral auction market subject to varying climatic conditions and policy constraints. We performed more than 15 experiments with university students and the general public in order to explore their behaviors in such an auction market.

We integrated all the aforementioned modules into the Middle Rio Grande Basin model and evaluated them by comparing them with historical data. We used these comparisons to calibrate the model and to explore the propagation of uncertainties in the context of a broadly integrated model. Finally, we documented our system dynamics toolbox in a SAND report written as a set of coordinated papers, which we will submit for publication in a peer-reviewed journal.

Significance

This project provides Sandia with a comprehensive resource management toolbox that:

- integrates the disparate systems of hydrology, ecology, climate, population, economics, policy, and water law into a coherent decision framework
- is fully generic, allowing application to a wide variety of watersheds spanning a broad range of scales
- operates within an interactive environment that allows decision makers and stakeholders to explore alternative water management decisions themselves.

The model has afforded business opportunities to Sandia's Water Initiative by providing a unique tool that aids resource managers at the local, state, and federal level. This project has helped spawn work in New Mexico, the US, and abroad. Specifically, new projects include:

- Middle Rio Grande Basin collaborative project, with the New Mexico Interstate Stream Commission, Bureau of Reclamation, US Geological Survey, and Army Corps of Engineers
- Gila Basin project, involving collaboration with the New Mexico Interstate Stream Commission, Bureau of Reclamation, Fish and Wildlife, and the Southwest New Mexico Water Planning Group
- Mimbres Basin Water Banking Study, with the Mimbres Water Users Group, New Mexico Office of the State Engineer, and the University of New Mexico
- Willamette Thermal Credit study, with the US Army Corps of Engineers Institute for Water Resources, Portland District Office, and the Willamette Partnership
- Zarqa River Basin Water Quality study, with the Jordanian Royal Scientific Society, Jordanian Ministry of Water and Irrigation, and the Jordan University of Science and Technology.

Additionally, Sandia, the Institute for Water Resources, and the US Geological Survey's Science Impact Centers are crafting efforts to develop a multiagency center for computer-aided dispute resolution (CADRe). CADRe's mission is to avoid or manage water conflicts through the application of computer-aided collaborative decision-making methods. It promotes the use of decision-support technologies within collaborative stakeholder processes to help stakeholders find common ground and create mutually beneficial water management solutions.

Its purpose also is to be a national leader in developing new methods and technologies that will help federal, state, and local water managers find innovative and balanced solutions to the nation's most vexing water problems. This toolbox project can contribute an important technical component toward the vitality of this fledgling center.

Refereed Communications

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Other Communications

V. Tidwell and H. Passell, "Cooperative Decision Support Modeling for Water Planning in the Middle Rio Grande," *Southwest Hydrology*, vol. 5, pp. 28-30, July 2006.

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D. Brookshire, D. Coursey, V. Tidwell, and C. Broadbent, "Water Banking and Leasing: Institutional Design and Integrated Modeling," in *Proceedings of the III International Symposium on Transboundary Waters Management*, July 2006, CD-ROM.

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D. Brookshire, C. Broadbent, V. Tidwell, and D. Coursey, "Towards a Water Leasing/Banking System on the Middle Rio Grande," presented at UCOWR/NIWR Annual Conference, Santa Fe, NM, July 2006.

J. Roach and V. Tidwell, "Groundwater-Surface Water Modeling in the Upper Rio Grande in Support of Policy Evaluation," presented at NGWA Groundwater Summit, San Antonio, TX, April 2006.

V. Tidwell, "System Dynamics Modeling to Support Community-Based Water Planning," presented at US Conference of Mayors Water Summit, Albuquerque, NM, September 2005.

V. Tidwell, "Interactive Planning Tool for the Upper Rio Grande," presented at UNM Utton Center Symposium on Rio Grande Reservoirs, Albuquerque, NM, April 2006.

V. Tidwell, "Integrated Decision-Support Modeling," presented at New Mexico Tech Graduate Seminar Series, Earth and Environmental Sciences Department, Socorro, NM, April 2006.

V. Tidwell, "System Dynamics Modeling to Support Community-Based Water Planning," presented at the University of Texas El Paso Graduate Seminar Series, Earth and Environmental Sciences Department, El Paso, TX, October 2005.

Predicting System Performance of Proton-Exchange Membrane Fuel Cells: Computational Modeling with Experimental Discovery and Validation

67053

K. S. Chen, N. P. Siegel, R. A. Assink, M. C. Celina, M. A. Hickner, D. R. Noble

Project Purpose

PEM (proton-exchange membrane, or polymer electrolyte membrane) fuel cells are emerging as a viable alternative clean power source for automotive and stationary applications. Before PEM fuel cells can be employed to power automobiles and homes, however, several key technical issues or challenges must be properly addressed.

One technical challenge is to elucidate the water distribution in, transport into, and removal from PEM fuel cells. Sufficient water is needed in the electrolyte or membrane to maintain sufficiently high proton conductivity, but too much liquid water present in the cathode can cause "flooding" (that is, pore space is filled with excessive liquid water) and hinder the transport of oxygen reactant from the gas flow channel to the three-phase reaction sites.

Another technical challenge is to understand the effects of nonuniform or anisotropic properties of the gas diffusion layer (GDL) on PEM fuel cell performance. The GDL anisotropy stems from the fact that GDLs used in PEM fuel cells are made from carbon paper or carbon cloth, which is characterized by its fibrous microstructure that gives rise to anisotropic transport properties. Yet another challenge is elucidating the membrane and membrane electrode assembly (MEA) degradation and failure mechanisms. Under practical operating conditions, fuel cell components such as the membrane and MEA degrade chemically and physically over time, creating a durability concern.

The purpose of the project is to address the aforementioned technical challenges; more specifically, it is to elucidate the key phenomena that control the performance and durability of PEM fuel cells by employing a combined approach of experimental investigation and computational modeling/analyses.

FY 2006 Accomplishments

In FY 2006, we focused our efforts on elucidating water distribution, transport, and removal; refining a finite-element model for simulating PEM fuel cell performance; and studying membrane and MEA degradation. We accomplished the following:

- Carried out additional and systematic neutron imaging experiments at the National Institute of Standards and Technology for probing liquid-water distribution within an operating PEM fuel cell and documented the findings in a refereed journal paper and a proceedings publication
- Refined and further developed a nonisothermal, two-phase model to study the anisotropic heat and mass transport in the GDL of PEM fuel cells and documented the effort in a proceedings paper and a manuscript
- Developed a two-phase flow model to simulate the two-phase pressure drop, liquid-water distribution, and flow maldistribution in PEM fuel cell channels and documented the effort in a manuscript
- Refined and further developed a finite-element model for simulating PEM fuel cell performance and documented the effort in a proceedings paper
- Carried out preliminary membrane degradation studies using nuclear magnetic resonance, oxidation rate (or oxygen consumption), and chemiluminescence techniques
- Performed an exploratory study on MEA degradation using transmission and scanning electron microscopy techniques.

Significance

The accomplishments in this project contribute to two core mission areas at Sandia: 1) science and technology and 2) energy security. In particular, efforts in this project have helped form “critical mass” at Sandia for conducting PEM fuel cell research and development and enabled Sandia to become a key player in the PEM fuel cell research community. In broader terms, accomplishments in this project can help Sandia to participate in the emerging hydrogen economy in which clean alternative automotive and stationary power generated by PEM fuel cells will play a critical role.

As a result of this project, we submitted a full proposal to DOE and participated in joint proposals (with Ballard Power Systems, Los Alamos National Laboratory, and University of Illinois at Urbana-Champaign) submitted to DOE in response to its special solicitation on fuel cell research and development. We also received funds-in from Ballard Power Systems to study water droplet instability.

Refereed Communications

M.A. Hickner, N.P. Siegel, K.S. Chen, D.N. McBrayer, D.S. Hussey, D.L. Jacobson, and M. Arif, “Real-Time Imaging of Liquid Water in an Operating Proton Exchange Membrane Fuel Cell,” *Journal of The Electrochemical Society*, vol. 153, pp. A902-A908, May 2006.

Other Communications

K.S. Chen and M.A. Hickner, “Modeling PEM Fuel Cell Performance Using the Finite-Element Method and a Fully-Coupled Implicit Solution Scheme via Newton’s Technique,” in *Proceedings of the ASME Fourth International Conference on Fuel Cell Science, Engineering and Technology*, p. 97032, June 2006.

U. Pasaogullari, P.P. Mukherjee, C.Y. Wang, and K.S. Chen, “Effect of Anisotropy of Gas Diffusion Layers on Two-Phase Heat and Mass Transfer in Polymer Electrolyte Fuel Cells,” to be published in *ECS Transactions*.

M.A. Hickner, N.P. Siegel, K.S. Chen, D.S. Hussey, D.L. Jacobson, and M. Arif, “Neutron Imaging Studies of Water and Heat Transport in PEM Fuel Cells,” to be published in *2006 Fuel Cell Seminar Conference Proceedings*.

Silicon Field Emission Electric Propulsion Arrays (FEEP) Powered by Orbital Nuclear Reactors

67055

R. X. Lenard, S. H. Kravitz, R. J. Shul, C. Schmidt, J. Nogan, J. G. Fleming, S. G. Rich

Project Purpose

The purpose of this project is to fabricate field emission electric propulsion (FEEP) microthrusters to demonstrate silicon-based thrusters powered by solar or nuclear energy. FEEPS are operational electric thrusters based on space-proven liquid-metal ion sources operating with indefinite lifetimes, high propellant efficiency (approaching 100 percent) and high electric efficiency (about 95 percent).

This year's objective was to complete the integration of all microfield emission thruster components and test the thruster at the Austrian Research Centers in Seibersdorf (ARCS).

FY 2006 Accomplishments

- Fabricated the emitters on 4-inch and 6-inch silicon wafers that had been prethinned to 500 microns.
- Bosch-etched a 5 x 5 pattern of holes that were 15 microns in diameter and 230 microns in depth.
- Steam reformed emitter holes to exhibit 10-micron diameter holes with a silicon dioxide liner.
- Deposited silicon nitride by chemical vapor deposition (CVD) to a depth of 2 microns, further reducing hole size.
- Deposited a 1-micron coat of tungsten to reveal a desired hole diameter of 4 microns.
- Designed extractors to be built from 100 micron thick pyrex blanks, etched conductive vias into those blanks, and sent them to Micronit (of the Netherlands) for abrasive machining.
- Successfully filled vias with copper for complete conduction pathways.
- Accomplished a flush-filled hole.
- Fabricated tank sides and end.
- Demonstrated anodic bonding of more than three layers (very significant accomplishment)
- Wet- and dry-etched emitters to expose tungsten coated silicon nitride emitter capillary.
- Investigated numerous wetting approaches, then designed and fabricated wetting booths. Based on final test results, it does not appear that the critical issue of getting indium to wet CVD'd tungsten was successfully resolved. During the project, we attempted chemical, mechanical, ion etching, gold-titanium precoat, and ion beam epitaxy methods with dubious results. Mechanical wetting appeared to generate the greatest promise. This is a critical-issue area that was never satisfactorily resolved.
- Fabricated all parts.
- Performed wetting and a microscopic analysis. Indium appeared to have wet tungsten.
- Bonded all parts together, some with epoxy, others anodically.

We packaged parts and prepared them for hand-carry to ARCS. Upon arrival at ARCS, several candidate parts were accepted for further testing. We inspected the contactor ring and modified the contact plate to be able to energize the extractor for emitter operation.

We placed the candidate thruster in the test chamber, applied power to melt the indium, and started the vacuum pump-down process. Additional melting was necessary, and the vacuum system was ready for operation. After the indium achieved the proper temperature, power was applied to the emitter. Voltage was increased until contact breakdown was observed.

Inspection later indicated that all of the emitter tips had been destroyed. Scanning electron microscope photos and digital photos were taken. Upon return to Sandia, we performed an energy dispersive x-ray analysis and determined that no indium was evident in the emitter deposits that occurred during operation.

Significance

Our project and findings are significant in four major aspects: integrated design, emitter fabrication, extractor design, and emitter wetting.

Integrated Design

Our team was the first to generate a fully integrated microelectromechanical systems (MEMS)-based design. The design allowed separate developments of each major component and produced the processes necessary to integrate all of the major components. One major first from these efforts was that we were the first team to anodically bond more than three layers simultaneously.

Emitter Fabrication

Other organizations have attempted to fabricate both the emitter and extractor segments employing microcircuit production techniques but have not been successful. Our team views as the primary issue the aspect ratio requirements for the restricted flow concept employed by ARCS. Their objective has been to mechanically restrict flow rates so that the problems that have plagued field emission thrusters in the past (large droplets of propellant in the ionization path), resulting in poor performance, can be resolved. The problem is that very long capillary length-to-diameter ratios are required; these aspect ratios are not achievable by conventional etching processes.

Our team used a combination of redefining the capillary length, hence diameter, and a very clever approach of etching a large hole first and filling it in with successive layers of material. This resulted in our team being the first to successfully develop a capillary with the desired characteristics. The fabrication process required many steps of masking, etching, back-filling, remasking, and etching to expose the capillary holes. Many steps of protective coating and etching that enabled varying rates of etch to properly reflect the design structure were developed and demonstrated. Alignment of the etching process to expose the capillary vias proved to be a particularly onerous issue, but one that was solved by very clever technicians on the team.

Extractor Design

The extractor design was also a first in that it exhibited separate electrical pathways for each emitter/extractor combination. We believe we have reduced this fabrication technique to a reasonably high degree of practice, and future developers should be well disposed in taking advantage of this fabrication technique.

Emitter Wetting

This is the only aspect of the project that was not completely successful. Wetting of the tungsten substrate with indium was essential for operation of the field emission thruster. We attempted many techniques, and although it appeared we had successfully demonstrated mechanical wetting, we were not able to extend this to chemical wetting. Failure to maintain wetting was demonstrated in testing.

Advanced Fuel Cell Reactor for the Direct Cogeneration of Electricity during Selective Partial Oxidation of Hydrocarbons

67056

A. H. McDaniel, S. F. Rice, R. E. Loehman, J. E. Miller

Project Purpose

A fuel cell operates on the principle that either hydrogen or hydrocarbon fuels can be completely oxidized to water and carbon dioxide while generating electricity. The idea of an advanced fuel cell reactor is to control oxidation in order to produce hydrogen or other valuable oxygenated hydrocarbons in addition to electricity. We propose an electrocatalytic membrane reactor that will make novel use of fossil fuels directly in solid oxide fuel cells (SOFCs) and at the same time address the broader needs of energy security by replacing the more conventional, less-efficient fuel reformer and selective oxidation processes.

We assembled a research team, focused on realizing these objectives, that combines materials and advanced diagnostics expertise. Activities include synthesis and evaluation of novel membrane electrode assemblies activated for selective oxidation of hydrocarbons. The project involves challenging materials development, catalyst formulation, and processing issues, as well as advanced diagnostics and experimentation.

The main objective is to prototype the reactor concept for a select class of partial oxidation reactions and thereby provide Sandia with a unique position in an area that has not been effectively exploited for SOFC applications. Unlike existing DOE programs targeting auxiliary power, the SOFC-like concept for combined chemical production and electrical generation is an emerging field.

FY 2006 Accomplishments

Acronyms:
SOFC = solid oxide fuel cell
Ni:YSZ = nickel mixed into polycrystalline yttria stabilized zirconia (50:50 wt%)
ScSZ = scandia stabilized zirconia
BHexa = barium hexa-aluminate ceramic material
POX, POM = partial oxidation, partial oxidation of methane
MEA = membrane electrode assembly

In the final project year, we continued to work on the thermo- and electrochemical characterization of the doped Ni:YSZ anode catalyst (dopants used were Ca, Mn, and BHexa) for POM to synthesis gas, and ethane dehydrogenation. In addition, we synthesized various ABO_3 perovskites of the form A = SrLa, BaLa and B = Co, Cu, or Co-Cu and characterized them for thermal POX of ethane, ethylene, propane, and propylene. We fabricated a gapped electrode design to allow us to observe bulk anode catalyst impedance during redox cycling in order to correlate the electrical properties of the thin films with chemical reactivity and selectivity.

This measurement has relevance to the SOFC community in that the current carrying properties of the anode catalyst must be sufficient to support electron transport from the triple-phase boundary region, and it is unknown how these properties vary with anode oxidation state. The team also successfully developed an in situ, high-temperature, pulsed Raman diagnostic capability and captured images of ScSZ electrolyte during redox cycling.

In a thermal sense, the doped Ni-based anode catalysts all behaved similarly in fuel-rich (high- equivalence ratio) environments, in that good conversion and selectivity to synthesis gas was observed for a short period of time, followed by a gradual decline in activity. None of the dopants appeared to have a positive effect on either delaying the onset of anode carbonization or stabilizing the nickel against oxidation. Both carbonization and oxidation tend to decrease catalyst activity and controlling this behavior is paramount to hardening cells for POX. Of real significance was the observed change in film impedance during redox cycling and POM, where the nature of the dopant appeared to influence this behavior as compared to a dopant-free Ni:YSZ material.

In the electrochemical sense, the original hypothesis regarding the chemical activity of membrane-transported oxygen species versus neutral, chemisorbed oxygen species has come into question. We originally proposed that the electrocatalytic behavior of the system could be tailored to promote conversion and selectivity by exploiting differences in reactivity between these two moieties; however, experiments on commercial MEAs proved otherwise.

We did not test the perovskites electrochemically because of difficulties in fabricating viable MEAs from these anode catalyst formulations. In fact, this project continually suffered from an inability to manufacture complex ceramic test fixtures. Of the dozen ABO_3 variants synthesized and tested, all showed similar activity toward ethane conversion and selectivity to ethylene with time-on-stream, which gradually degraded to higher CO selectivity, signaling deactivation presumably through formation of lanthanum oxide phases.

Catalysts containing either Co alone or Cu alone on the B cation sites performed better than catalysts containing both Co and Cu. In addition, the partial substitution of La by Ba was preferable to Sr. Near the project's end, we suspended two perovskite formulations over platinum-and gold-patterned electrolytes to investigate control of the membrane-transported oxygen as a means to moderate thermal POX (which is different than electrochemical POX).

Significance

The SOFC scientific community at large would be interested in our observations made on doped Ni:YSZ anode catalysts in two regards: (1) the effects of additives intended to stabilize the anode against carbonization and oxidation, and (2) bulk electrical properties of these doped thin films.

At present, researchers are still looking to make SOFC systems more fuel flexible, and understanding how to change the anode catalyst formulations is key to maintaining the desired reactivity (promoting desirable reactions while inhibiting others) and electrical conductivity. A journal article documenting these results is in preparation.

Ultimately, the farther reaching goals of this project were compromised by material fabrication issues; however, this effort did establish a viable research capability in the field of high-temperature membrane reactor science, a capability that is well aligned with alternative strategies for creating more energy-efficient chemical processes. We anticipate that this capability will be leveraged by future Sandia work for DOE/Basic Energy Sciences programs.

Membranes for H_2 Generation from Nuclear-Powered Thermochemical Cycles

67059

T. M. Nenoff, K. Leung, A. Ambrosini, T. J. Garino, F. Gelbard

Project Purpose

Efficient and environmentally friendly methods for producing hydrogen are important as the world explores the use of hydrogen as a clean energy source. One such method is the production of hydrogen via high-temperature thermochemical cycles driven by heat from solar energy or next-generation nuclear power plants. These processes are advantageous because they do not produce greenhouse gas emissions that result from hydrogen production by electrolysis (using fossil fuel electricity sources) or hydrocarbon reformation. Based on a previous extensive survey of more than 100 thermochemical cycles, which evaluated metrics such as efficiency, cost, number of steps, and the number and phases of reactants, the sulfur-iodine (SI) cycle was deemed to be one of the most promising.

The first step of the SI cycle involves the decomposition of H_2SO_4 into O_2, SO_2, and H_2O at temperatures around 850 ºC. In situ removal of O_2 from this reaction pushes the equilibrium toward dissociation, thus increasing the overall efficiency of the decomposition reaction and possibly lowering the temperature required to achieve this step. The membrane required for this oxygen separation step must withstand the high temperatures and corrosive conditions inherent in this process.

Perovskites and perovskite-related structures are potential materials for oxygen separation membranes owing to their robustness, ability to form dense ceramics, capacity to stabilize oxygen non-stoichiometry, and mixed ionic/electronic conductivity. A mixed ionic-electronic conductor benefits the process by allowing the separation of oxygen via ionic conductivity, while the electronic conductivity eliminates the need for an applied potential across the membrane to balance the oxygen ion flux.

To this end, we undertook a survey (literature and experimental) of potential perovskite and related compounds. We synthesized powder samples by solid-state methods and characterized them using powder x-ray diffraction (XRD), thermogravimetric analysis (TGA), and elemental analysis. We used TGA versus temperature and oxygen partial pressure as a screening method to test the oxygen absorption and desorption properties and stability of the materials.

Two oxide families gave promising initial results, and we decided to study them further. The families are the double-substituted perovskite $A_xSr_{1-x}Co_{1-y}B_yO_{3-d}$ (A=La, Y; B=Cr, Ni) – in particular the family $La_xSr_{1-x}Co_{1-y}Mn_yO_{3-d}$ (LSCM) – and doped La_2NiO_4.

We synthesized circular, 12 mm diameter self-supported membranes of these materials by isostatic pressing and sintering the powders and characterized them by XRD, four-probe conductivity, and scanning electron microscopy. We constructed a high-temperature oxygen permeation unit for oxygen permeability measurements at 850 ºC, and took additional permeation measurements on the LSCM materials in collaboration with Eltron Research, Inc. In addition, we placed materials in a H_2SO_4 decomposition reactor to test for stability under high temperature and corrosive conditions.

FY 2006 Accomplishments

We placed samples of LSCM and doped La_2NiO_4 in a H_2SO_4 decomposition reactor, and the nickelate samples decomposed almost completely. Qualitative analysis of LSCM by scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS) showed corrosion on the surface of the pellet, extending several microns, but the membrane itself remained relative intact. The major corrosion was found to be $SrSO_4$.

Preliminary permeation measurements by Eltron show that the LSCM materials are oxygen permeable between the temperatures of 800-950 ºC, but further permeation measurements on the LSCM materials have been hindered by the inability to achieve densities greater than 95 percent of theoretical. As a result, gas is able to transport through the micropores present in the sintered membranes. Through collaboration with Eltron, we have been able to improve densities and are nearing the desired goal. We are also currently working on getting the in-house permeation unit operational.

Conductivity experiments versus temperature and pO_2 on LSCM materials show the materials are mixed ionic-electronic conductors. The overall conductivities are on the order of 101 S/cm. The ionic contribution to the conductivity is estimated on the order of 10^{-1} S/cm.

Attempted structural refinement of the LSCM1973 phase by XRD and TEM implies that the material crystallizes in the cubic Pm-3m space group. However, further investigation using neutron diffraction (by Hongwu Xu at the Los Alamos Neutron Science Center) implies that a slight crystallographic distortion may be present.

We performed density functional theory (i.e., electronic structure) calculations for the end member material $SrCoO_{3-d}$ as well as $La_{0.125}Sr_{0.875}Co_{0.875}Mn_{0.125}O_{3-d}$. We used supercells (i.e., replications of the basic formula unit cubic primitive cell) of size 2x2x2 containing 40 atoms; d=0 or 0.042, corresponding +/- one oxygen vacancy. Using both LDA and LDA+U local density approximation methods, all materials are found to exhibit a magnetic moment exceeding 2 Bohr magnetron and zero band gap. The oxygen vacancy hopping barrier is small, on the order of 0.30-0.58 eV. The La/Mn doped oxide exhibits the smallest barrier to vacancy motion, in apparent agreement with experimental measurements of ionic conductivity. These barriers are extremely small compared to the ~ 2 eV barriers found in insulating perovskite oxides such as potassium tantalate.

We completed calorimetry for the LSCM samples and for the cobalt end member $La_{0.1}Sr_{0.9}CoO_3$ (LSC 191). Iodometric titration of LSC 191 gave an average delta value of 0.29 +/- 0.01. Based on the microprobe analysis of the sample, a composition was determined of $La_{0.1}Sr_{0.96}Co_{1.07}O_{2.72}$ that is consistent with the observed oxygen content and a charge of 3^+ for Co. In order to find a trend in the observed enthalpies of formation from oxides for the three LSCM compositions, we calculated an average oxidation state (AOS) of Mn for 10 ratios of $[Co^{3+}/(Co^{3+} + Co^{4+})]$. We observed that for a ratio of 0.6 (this ratio is unique), there is an almost linear correspondence in the AOS of Mn, with respect to the enthalpies of formation.

Significance

Successful development of high-temperature oxygen separation membranes would advance the goals for hydrogen production via thermochemical cycles by increasing the efficiency and lowering the reaction temperature of the sulfuric acid decomposition step of the SI cycle. This would increase the net energy output of the SI cycle, which is essential to this cycle becoming a practical method of hydrogen production. This work can be leveraged into the larger DOE Nuclear Energy program on thermochemical cycles as a starting point for more optimized ceramic-based membranes. Preliminary results have been promising enough to secure funding for follow-on work from the DOE Office of Nuclear Energy.

Refereed Communications

A. Ambrosini, T. Garino, and T.M. Nenoff, "Synthesis and Characterization of the Double-Substituted Perovskites $La_xSr_{1-x}Co_{1-y}Mn_yO_{3-d}$ for Use in High-Temperature Oxygen Separations," to be published in *Solid State Ionics*.

Other Communications

A. Ambrosini, T. Garino, T.M. Nenoff, R.G. Iyer, A. Navrotsky, and H. Xu, "Synthesis, Characterization, and Permeation Properties of $La_xSr_{1-x}Co_{1-y}Mn_yO_{3-d}$ Oxygen Separation Membranes," presented at the Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2005.

A. Ambrosini, "Synthesis and Characterization of Perovskite-Based Oxygen Separation Membranes," presented (invited) at Laboratoire CRISMAT, Caen, France, October 2005.

Tunable Ion Conductors for Low-Temperature Oxide-Based Fuel Cells

67061

T. M. Nenoff, T. J. Garino, J. D. Pless

Project Purpose

The purpose of our project was to discover new ion-conducting, solid-phase materials that have better conductivity values at temperatures between 550-800 °C than the industry standard yttrium stabilized zirconium (YSZ). We also wanted to understand the structure/property relationship between nanoscale manipulation of ceramic phases, ABO_3 perovskite structures, and their bulk conductivity responses with temperature. We were successful in developing the new method of synthesis, expanding the phase-space of perovskites into the niobate phases, and achieving an order of magnitude better ion conductivity than YSZ.

The basic research behind the synthesis and characterization led to a structure/property relationship theory that we used to direct research. In it we discovered that substitution on the A-site of the ABO_3 perovskites had a direct effect on the bulk conductivity. The size of cations substituted onto that site was directly proportional to the conductivity; the smaller the cation (e.g., Mg^{2+}), the higher the conductivity.

FY 2006 Accomplishments

- We completed conductivity and calorimetric studies of all new phases synthesized in the project.
- We formulated a predictive model of the structure/property relationship for the metastable phases.
- We finalized our "best" material formulation of Mg/Nb/Ti/O perovskite.
- We submitted a technical advance (SD-10336) regarding similar materials for photocatalysis.

Significance

The results are of importance to Sandia and DOE because ion conductors are used in solid oxide fuel cells and as catalysts. In addition, these perovskites are used as waste storage materials and are being leveraged by DOE Office of Nuclear Energy. As a result of this project, we established industrial collaborations for follow-on work. We also submitted a proposal, with an industrial collaborator, to the DOE Nuclear Energy Research Initiative for follow-on work in FY 2007-2009.

Refereed Communications

J.D. Pless, R.G. Iyer, J. Maslar, T. Garino, A. Navrotsky, and T.M. Nenoff, "Structure-Property Relationships of Tunable Ionic Conductivity in Niobate Perovskites," to be published in *JACS*.

Critical Infrastructure System of Systems Assessment Methodology

67114

J. M. Phelan, J. E. Stamp, G. D. Wyss, J. J. Danneels, P. L. Campbell, T. M. Torres, G. B. Varnado, R. C. Parks

Project Purpose

Protecting critical infrastructure facilities against malevolent attacks is a major challenge for facility operators. Traditional security threats from vandals seeking to deface property or cause inconsequential damage have been managed by basic security principles, such as perimeter protection and periodic surveillance. However, an emerging adversary with a philosophical intent to destroy American society may consider more sophisticated attacks to cause widespread damage to critical infrastructures. As critical infrastructure business practices leverage more system automation, security assessment technology must also be able to evaluate the relationship between cyber and physical security and its implications for unidentified vulnerabilities.

Most of the critical infrastructures deliver a commodity such as water, power, or natural gas to an end user. An infrastructure facility comprises "assets," such as systems, subsystems, or components that must operate properly in order for the facility to perform its intended function. The facilities include commodity-delivery assets (e.g., pumps, valves, and transformers) and cyber elements that can control set-points, actuation, or other operating functions for the commodity-delivery assets.

The facilities will have some form of physical protection (e.g., fences, locks, and alarm systems) and some form of cyber protection (e.g., firewalls and administrative access controls). Some of the physical protection system (PPS) elements may be controlled or monitored by cyber means. Interactions between physical and cyber security are recognized in the popular media (such as in the Oceans 11 movie) and the security community. However, most risk and vulnerability assessment research has focused on either physical or cyber security.

This effort first explored various historical approaches to physical and cyber security assessment methods. Upon finding no satisfactory existing method that considered both physical and cyber attacks, we developed a new approach to evaluate "blended attacks," where the adversary makes use of both physical and cyber attack tactics.

The purpose of this project was to develop a risk assessment methodology that supports analysis of integrated physical and cyber security elements within critical infrastructure systems. We also wanted to achieve a better understanding of these cyber/physical interfaces and their implications for unidentified vulnerabilities and to provide decision makers with integrated and comprehensive risk results for "blended" security systems that contain both cyber and physical elements.

FY 2006 Accomplishments

Security assessment methods, engineering failure analysis methods, and risk assessment methods all have similar purposes that seek a better understanding of system failures that cause significant consequences. Physical-security assessment methods and cyber-security assessment methods also share this same purpose. However, differing historic approaches to achieve these purposes provided significant challenges to develop a risk assessment methodology that supports analysis of integrated physical and cyber security elements within critical infrastructure systems.

Analytic methods for engineering failure analysis provide a foundation to determine which system components and sequences of events must fail to cause a specific consequence. Yet, security assessments require additional steps to understand whether an adversary capability can overcome security systems that protect critical components. Physical security analysis uses a “detect, delay, and respond” method to evaluate physical system effectiveness; however, cyber security analysis typically uses “best practices” or “red team engagements” to evaluate cyber system effectiveness.

When path-based approaches (e.g., attack graphs) are used to evaluate cyber protection systems, those analyses typically lack the quantitative details found in PPS analyses. Risk analysis requires integration of consequence and vulnerability estimates to gauge the potential impacts to critical infrastructure. Integrating select aspects from each of these methods was the key to achieving the project goal.

We waded through the historic approaches, and retained valuable aspects and tabled marginal ones, in order to develop a truly integrated cyber/physical security assessment methodology. The most important outcomes of this work are that:

- We achieved a better understanding of the cyber/physical interfaces and implications for unidentified vulnerabilities, and
- We developed a tool for decision makers that shows integrated and comprehensive risk results for “blended” security systems that can contain both cyber and physical elements.

The results of our efforts were two distinct cyber-physical security assessment tools for targeting differing levels of security system sophistication. For less sophisticated security systems, we developed a best-practices-based assessment tool that uses a questionnaire format with links to both cyber and physical security best practices. For more sophisticated security systems, we developed a functional assessment methodology that evaluates combined cyber and physical security within a risk management approach.

Significance

We recognized that not all security systems are of similar sophistication, nor should they be. Security systems for low-consequence impacts, or where mitigation might provide adequate risk management, could be evaluated with a best-practices or screening-analysis method. Our best practices questionnaire analysis tool (CICSTART) evaluates both physical and cyber security practices. Conversely, high-consequence impacts or difficult to mitigate risks often have more sophisticated security systems, which require functional or engagement style security systems analysis. Our functional style security assessment method (CPSAM) integrates cyber and physical security systems as a software application.

The CPSAM functional risk assessment methodology combines the fundamentals of physical protection systems (e.g., detect, delay, and respond) with cyber protection system primitives that are based on opportunistic pathway analysis. The methodology begins with a fundamental risk principle where the analysis selects specific consequences of concern so resources are not wasted looking at inconsequential impacts. Specific key asset failures that could lead to those consequences are identified by external analysis methods, as these are often tailored to the complexities of the specific infrastructure.

The capabilities of the adversary attacking the facility are contrasted with the protective features at the facility to estimate the likelihood of adversary success. The key cyber-physical security integration step occurs in the portion of the vulnerability assessment model, where the performance of protection elements that are cyber-controlled are turned off to account for the likelihood that an attacker could penetrate the cyber protection system.

Since there is insufficient data to support a probabilistic approach to cyber security assessment, we developed a novel application of a broader mathematical tool (belief and plausibility) to support vulnerability estimates for attacks that include cyber elements.

Refereed Communications

J. Darby, J. Phelan, P. Sholander, B. Smith, A. Walter, and G. Wyss, “Evidence-Based Techniques for Evaluating Cyber Protection Systems for Critical Infrastructures,” in *Proceedings of the MILCOM 2006*, October 2006, CD-ROM

Nanoscale Optical and Electrical Probes of Materials and Processes

79776

K. H. Bogart, M. H. Crawford, C. H. Fujimoto, S. R. Kurtz, M. A. Quintana

Project Purpose

Near-field scanning optical microscopy (NSOM) has the potential to integrate optical and scanning probe microscopy (SPM) techniques in order to generate optical, electrical, and structural images with nanometer-scale resolution. We are developing new measurements around an NSOM-SPM instrument that is unique in its ability to capture electrical and morphological information concurrently with optical spectra, spanning the 325-1100 nm wavelength range. We are applying this novel tool to energy conversion device technologies, specifically III-nitride-based solid-state lighting, light-emitting diodes (LEDs), II-VI-based solar cells, and polymers for organic LEDs (OLEDs).

Inhomogeneities in epitaxial materials are sources for undesirable physical mechanisms that limit device efficiencies. The specific effect of these imperfections (dislocations, defects, impurities, segregations) on the optical and electrical properties of semiconductor materials is not well understood. For example, optical emission is believed to occur preferentially in indium-rich regions formed by phase separation in InGaN. Concurrent optical, electrical, and morphological measurements of these imperfections are limited by instrument incompatibilities and by the submicrometer size of these features.

Until recently, NSOM and electrical SPM with nanometer-resolution have not been available on-site for Sandia's semiconductor material and device research. Our dedicated instrument has the capability to perform high-resolution (100 nm) NSOM and SPM measurements concurrently, as well as lower-resolution (~ 1 µm) optical confocal measurements, and is unique with optics extending into the ultraviolet (325 nm) and with robust NSOM apertures based on Al-coated Si/Si_3N_4 atomic force microscope probe tips.

We will create confocal- and NSOM-based, spectrally-resolved photoluminescence spatial images of InGaN quantum wells under development as blue/green LEDs for solid state lighting. We will endeavor to spatially resolve (< 1 µm confocal and < 100 nm NSOM) regions of indium segregation and identify the growth conditions to increase emission efficiencies. Alternatively, comparison of photocurrent and morphological images of these LEDs can be used to identify dislocations and other features which control minority carrier lifetime and determine device performance.

We will compare high-resolution photocurrent and morphological images of LEDs and solar cells to identify dislocations and other features that affect minority carrier lifetime and determine device performance. Similarly, we will try to observe localized defects in solar cells with photocurrent images or optimize surface texturing used to enhance solar cell efficiencies. We will also perform photoluminescence studies of polymeric thin films for OLEDs to determine emission properties, relative intensities, and wavelengths of these materials.

FY 2006 Accomplishments

During FY 2006, we improved the performance of the NSOM system. We determined the ultraviolet optics train in our system was severely degraded and replaced the optics. We also upgraded one of the charge-coupled device cameras, improving detection of laser signal during system alignment, and upgraded the collection software package, increasing confocal-mode capabilities.

We determined that InGaN quantum wells grown by metal-organic chemical vapor deposition over GaN films containing large-scale (200-40,000 nm) physical defects such as pits or ridges emit photo-luminescence at shorter wavelengths than expected for the amount of indium in the InGaN material. The source of the physical defects can be from nonideal coalescence of nucleation islands in the underlying GaN or can be from v-defect formation (pits) from threading dislocations originating in the underlying GaN. We believe that as the quantum well is deposited over the defect, the thickness of the quantum well decreases along the sloped surface of the defect. Thinner quantum wells induce emission at shorter wavelengths by an increase in the bandgap.

We also determined that for GaN/InGaN-related materials analyses, we need a second laser of longer wavelength (405 nm) to induce resonant photoluminescence emission in InGaN, thus eliminating emission from the underlying GaN materials.

We also contributed to the understanding of photoluminescence of thin film, spun-cast organic polymers for OLEDs.

Significance

We were able to establish a new materials analysis capability at Sandia that combines micrometer- and nanometer-scale resolution of photoluminescence, spectrally-resolved photoluminescence, and scanning probe microscopies such as atomic force microscopy. This project helped us to make our Sandia colleagues aware of this capability and enabled us to use our new materials analysis system to contribute to other Sandia projects, including LDRD project 102613, “Phase Imprint Lithography for Large Area 3D Nanostructures,” and LDRD project 102600, “Nanoengineering for Solid-State Lighting.”

Applying New Network Security Technologies to SCADA Systems

79777

S. A. Hurd, J. Margulies, D. P. Duggan, P. L. Campbell, A. Berry, A. R. Chavez, D. Kilman, J. E. Stamp

Project Purpose

Supervisory control and data acquisition (SCADA) systems for automation are important for critical infrastructure and manufacturing operations. SCADA systems collect and transmit information between sensors, controllers, and central management stations, and concurrently store, process, and analyze that information. They work in a number of physical environments using a variety of hardware, software, networking protocols, and communications technologies.

One common thread among SCADA systems is they were developed without adequate regard for security issues. Many legacy SCADA systems were implemented in an era before security issues became a major concern. Even for contemporary SCADA technology, the most important factors appear to be the lack of a perceived business case for SCADA security features, and the reluctance of SCADA operators to implement security features that might impede the operation of the SCADA system.

Research at Sandia and elsewhere has focused on how to improve security and reliability for next-generation SCADA systems over the long term. However, as these systems are often attractive targets for adversaries, there is a critical need to identify ways to address these security shortcomings in the short and medium term.

We will investigate how technologies developed to secure conventional information technology (IT) networks could be applied to address securing SCADA systems and networks. We can accomplish this by bringing together:

- Some of the world's best operational network defense and intrusion detection analysts
- Intrusion detection, adaptive network countermeasures, and encryption researchers
- SCADA security assessment experts.

Our endgoal is to identify and test technologies that will significantly improve the security posture of existing SCADA systems and that are simple to implement, reliable, and acceptable to SCADA owners and operators.

FY 2006 Accomplishments

- We designed a standard architecture for add-on security devices for SCADA systems based upon conventional IT approaches (primarily open-source approaches).
- We built and configured prototype add-on security devices for SCADA systems.
- We tested the prototype add-on security devices for efficacy (e.g., added security functionality, acceptably small introduction of latency, and so on). The devices had the desired security functionality and introduced latency that was well within system tolerance levels.
- We tested the prototype add-on security devices for security vulnerabilities. A red team discovered a potentially serious misconfiguration (which was easily fixed and noted for future revisions). Afterwards, the red team found no glaring security problems with the add-on devices.

Significance

The primary significance of our work is that we demonstrated that relatively low-cost add-on security devices for SCADA systems (primarily using existing open-source components) can work effectively, reliably, and with negligible performance affect on existing SCADA systems. In particular, we:

- Demonstrated add-on security devices to DOE, which then funded follow-on work for further development, testing, and technology transfer of these devices
- Demonstrated add-on security devices to industry representatives. As a result, an industry advisory panel was formed to guide further development and to encourage technology transfer.

In addition, our project highlighted the additional work necessary to allow for wide-scale production system deployment of these devices. Primarily, the additional work will be needed in device configuration and management.

Finally, the work energized the DOE, as well as the industry and vendor communities, regarding the use of add-on security devices for SCADA systems. There had been significant speculation that such devices, along with the work to develop such devices, would be helpful. However, previous work had focused exclusively on proprietary, rather than open-source solutions.

Since our initial presentations on this work, numerous industry participants have expressed interest in testing these devices. Furthermore, numerous vendors have expressed interest in making this technology available commercially to industry (or developing and selling similar technology).

Other Communications

J. E. Stamp, "SCADA Linux APpliance: SLAP System," presented at Clemson Power Systems Workshop, Clemson, SC, November 2005.

J. E. Stamp, "SCADA Linux APpliance: SLAP System," presented at DOE National SCADA Test Bed Leadership Meeting, Washington, DC, March 2006.

J. E. Stamp, "SCADA Linux APpliance: SLAP System," presented at Clemson Power System Conference, Clemson, SC, March 2006.

J. E. Stamp, "Situational Awareness in Process Control Networks," presented at The Institute for Information Infrastructure Protection (I3P), San Diego, CA, June 2006.

Use of Composite Materials to Refurbish Our Civil and Military Infrastructure

79778

D. P. Roach, E. D. Reedy Jr., K. A. Rackow, W. A. Delong, K. B. Pfeifer, E. Yepez

Project Purpose

The goal of this project is to establish bonded composite doublers as a reliable and cost-effective structural repair method for civil and military structures and to develop adequate real-time monitoring and self-healing systems to ensure the long-term integrity of such structures with minimal need for human intervention. To accomplish these objectives, we coupled prototype installations testing with theoretical modeling in a four-element approach:

- design and performance of composite repairs
- in situ sensors for real-time health monitoring
- self-healing of in-service damage in a repair
- numerical modeling

Economic barriers to the replacement of steel structures have created an aging infrastructure and placed even greater demands on efficient and safe repair methods. Furthermore, as a result of homeland security concerns, increased attention has been placed on the rapid repair and pre-emptive reinforcement of buildings and bridges. This project deals with the repair or reinforcement of steel structures using high-modulus fiber-reinforced polymer (FRP) material.

The use of bonded composite doublers has the potential to correct the problems associated with current repair techniques and has the ability to be applied where there are no rehabilitation options. It promises to be cost-effective with minimal disruption to the users of the structure. Instead of fastening multiple steel plates to implement a repair, it should be possible to bond a single FRP composite doubler to the damaged structure.

When considering the military applications, it should be noted that rapid repair objectives are inherent in this project so that this technology will allow for quick return-to-service of military structures and vehicles through the rapid repair of battle damage. In the matter of bridge refurbishment alone, the National Bridge Inventory Database (Federal Highway Administration, 2003) indicates that 30 percent of the 591,000 bridges in the US are "structurally deficient."

FY 2006 Accomplishments

Design, Installation, and Performance Assessment of Composite Repairs

Using fatigue and ultimate strength tests to evaluate the overall effect of composite doublers on stress intensity, crack growth, and strength characteristics of steel structure, we were able to determine the potential for disbonds between the steel and the laminate, the crack mitigation capabilities of composite doublers in the presence of severe defects, and an optimum composite doubler design philosophy.

Results show that the fatigue life of steel structures can be extended by a factor of 10 through the use of composite patches. Residual strength failure tests demonstrated that boron-epoxy doublers are able to return the parent structure to its original strength and load-carrying capability.

Smart Structures and Health Monitoring Sensors

We conducted performance testing to evaluate the health monitoring capabilities of fiber optic (FOBG) and piezoelectric (PZT) sensors and to optimize their design. The PZT and FOBG networks and data analysis software detected and properly sized fatigue cracks and disbonds in composite repair specimens.

In the eddy current sensor arena, we developed a more sophisticated electronics package – including digital signal processing to filter and detect phase shifts – and improved the dual coil configuration to further improve probe sensitivity. We built the electronics system to accommodate self-calibration, data acquisition, digital signal processing, diagnostics, and data storage/transmission. We achieved excellent crack detection even when inspecting through thick composite repairs.

Self-Healing in the Composite Laminate Repair
We optimized the healing agent encapsulation process to minimize the capsule size while producing reliable release of the healing agents and stable suspension of the catalyst in the resin. The investigation determined the concentration of capsules needed for self-healing without negatively affecting the strength of the original adhesive.

We also investigated deployment of the capsules in the adhesive film to create a fully self-healing adhesive; healing in the presence of continued stress; and the performance and efficiency of self-healing laminates through strength, fracture, and fatigue testing of baseline and self-healed composite specimens. In addition, we identified a healing system that uses dicyclopentadiene monomer as the microencapsulated healing agent coupled with an embedded Grubbs catalyst. Successful healing was accomplished by incorporating a small volume of microcapsules in an epoxy resin along with the self-healing catalysts.

Numerical Modeling
We developed finite element models in order to produce an understanding of the critical elements of this technology and to develop design guidelines with an associated software package to aid the design process. By including models with and without the composite doubler present, we could study the overall effectiveness of each composite repair design feature. We used cohesive zone elements to simulate steel crack and composite disbonds. We validated the finite element models by comparing simulation results to the strain mapping and flaw growth data produced via experimental structural tests.

Design Analysis Software
We produced the initial version of the design analysis tool for implementation in a spreadsheet.

Significance

Composite materials and smart structures can offer significant benefits in DOE applications, including weapons, energy and critical infrastructure, homeland security, transportation, and manufacturing. Their use has been limited by uncertainties in their application and by an inability to assess structural integrity, provide preventive health monitoring, and anticipate failure. This project enables more widespread deployment of advanced composite materials to improve performance and reduce cost.

Similarly, advances in nondestructive inspection methods and related structural health-monitoring sensors can be applied in a wide variety of DOE missions. We developed customized sensors and in situ structural health-monitoring approaches. Distributed sensor networks can now be viewed as viable options to address surveillance, security, and structural integrity needs. Unique sensors, when considered in concert with self-healing methodology, produce smart or adaptive structures that are key elements in the general science and technology community as well as building blocks in infrastructure (e.g., bridges, buildings, aircraft, and satellites) and a myriad of mechanical systems.

Finally, numerical modeling activities give rise to general scientific advancements (e.g., modeling of cracks and other flaws) and modifications to Sandia codes to accommodate composite models. These developments can be applied across many Sandia numerical analysis programs. Efforts are already under way to leverage these capabilities in health-monitoring applications (DOE weapons, National Aeronautic and Space Administration vehicles, Navy ships, Air Force aircraft) and composite design and analysis work (Boeing, Department of Transportation, Federal Aviation Administration).

Refereed Communications

A.H. Kumar, D.P. Roach, and R.A. Hanum, "In Situ Monitoring of the Integrity of Bonded Repair Patches on Civil Infrastructures," in *Proceedings of the SPIE Smart Structures and Materials Symposium*, p. 12, February 2006.

D.P. Roach, J.M. Kollgaard, and S.A. Emery, "Application and Certification of Comparative Vacuum Monitoring Sensors for In Situ Crack Detection," in *Proceedings of the Air Transport Association Nondestructive Testing Forum*, p. 22, September 2006.

D.P. Roach, "Health Monitoring of Aircraft Structures Using Distributed Sensor Systems," in *Proceedings of the DoD/NASA/FAA Aging Aircraft Conference*, p. 31, March 2006.

J.R. Zayas and D.P. Roach, "Low-Cost Fiber Bragg Grating Interrogation System for In Situ Assessment of Structures," to be published in *Proceedings of SPIE Conference on Nondestructive Evaluation for Health Monitoring and Diagnostics*.

Other Communications

D.P. Roach, "Can Wind Turbine Blade Designs Benefit from the Application of Aircraft Health Monitoring and Advanced Materials Research?" in *Proceedings of the Sandia National Laboratories 2nd Wind Turbine Blade Workshop*, p. 25, April 2006.

D.P. Roach, "Application of Aerospace Health and Usage Monitoring Technology to Wind Plant Operations," in *Proceedings of the Integrated Wind Plant Predictive Maintenance Management Workshop*, p. 18, April 2006.

Desalination Utilizing Clathrate Hydrates

79779

B. A. Simmons, C. L. Staiger, J. A. Greathouse, W. A. Anderson, R. W. Bradshaw, R. T. Cygan, D. E. Dedrick, E. H. Majzoub

Project Purpose

The cheap and efficient production of plentiful fresh water is becoming increasingly critical to the sustainability and security of our national infrastructure. Present desalination methods, such as high-temperature distillation, reverse osmosis, and/or electrodialysis, can purify water but suffer from certain limitations.

We are developing a technology that achieves water desalination by harnessing and optimizing clathrate hydrate phenomena. Clathrate hydrates are crystalline compounds of gas and water that desalinate water by excluding salt molecules during crystallization. Contacting a hydrate-forming gaseous species with water will spontaneously form hydrates at specific temperatures and pressures through the extraction of water molecules from the bulk phase followed by crystallite nucleation. Subsequent dissociation of pure hydrates yields fresh water and, if operated correctly, allows the hydrate-forming gas to be efficiently recycled into the process stream.

Investigations of clathrate hydrates as a means to desalinate water have met with mixed results. The major obstacles encountered were identifying the appropriate hydrate formers and the efficient removal of excess salt that remained from the original solutes and/or was entrapped in the formed hydrate.

We are investigating a novel approach that may solve these problems by the use of novel refrigerant hydrate formers and a bulk inert heat exchange liquid that uses the different physical properties of the crystals (hydrate and solute) formed and the chemical species used in the nucleation process. This will result in a process by which the hydrates will preferentially separate from the salt crystals through the precise control of nucleation and/or the specific hydrate-forming environment and bulk phase properties. We are using both experimental batch-scale results and the data generated from computational modeling of these hydrates to develop a system that can efficiently and reliably desalinate water from virtually any type of input source.

FY 2006 Accomplishments

We exceeded the FY 2006 project milestones. In FY 2005 we developed, built, and used a commercially available system that allowed for batch desalination. This year, we moved into the realm of determining the impact of numerous salt concentrations on the kinetics of hydrate formation, pressure-temperature equilibria determination, and hydrate stability observations.

To accomplish our objectives, we fabricated and/or modified two existing commercial reactors that had multiple connectors to allow for fluidic inlets and outlets, pressure and temperature monitoring, stirring, active controlled cooling, and real-time optical and data recording. We formed multiple hydrate species in these reactors and characterized them with x-ray diffraction and Raman spectrometry. Our results are in agreement with the data published on these systems. We also established the link between the computational effort and the experimental results, which will enable the overall successful completion of the project in FY 2007.

Significance

The realization of a robust, efficient, and economic means of producing fresh water is a critical component in the overall national security portfolio. The nation's water supply has been of great interest and is one of the main components of DOE's mission in terms of national security. The desalination of water through clathrate hydrate formation is an economically and energetically desirable option that has been brought forward as a viable means of adding to this technology portfolio.

This project has generated a significant contribution to the field of hydrate desalination. The computational results, in particular, will have far-reaching impact outside the field of desalination in clathrate hydrate phenomena.

We presented this work at a special session of the 2006 Spring Materials Research Society Conference on desalination technology, published a peer-reviewed paper in the *Journal of Physical Chemistry* on the computational modeling effort, have a patent application on the technique of using a heat exchange liquid under revision, and have another two manuscripts in preparation for submission to peer-reviewed journals.

Refereed Communications

J.A. Greathouse, R.T. Cygan, and B.A. Simmons, "Vibrational Spectra of Methane Clathrate Hydrates from Molecular Dynamics Simulation," *Journal of Physical Chemistry B*, vol. 110(13), pp. 6428-6431, May 2006.

Other Communications

R.W. Bradshaw, B.A. Simmons, E.H. Majzoub, W.M. Clift, and D.E. Dedrick, "Clathrate Hydrates for Production of Potable Water," to be published in *Proceedings of the MRS National Spring Meeting*.

Development and Application of the Dynamic System Doctor to Nuclear Reactor Probabilistic Risk Assessments

79780

D. M. Kunsman, S. Dunagan

Project Purpose

The Dynamic System Doctor (DSD) is a system-independent multitask driver (MTD) under development at Ohio State University for model-based state/parameter estimation in dynamic systems. It can provide input to the real-time analysis for evolving systems conditions and has been successfully applied to real-time xenon estimation in a reactor, stability analysis for a boiled water reactor, and fault detection in automobile engines. The DSD has also been linked with a neural net approach.

The purpose of this project is to link the DSD to MELCOR so that it directly and semiautomatically helps us construct additional tree logic. MELCOR is a detailed thermal/hydraulic severe accident code developed at Sandia. We thought the DSD would link directly to the accident progression event tree (APET), which then would link to the MELCOR model. We found, however, that it is more efficient to link the driver directly to MELCOR from the perspective of an overall probabilistic risk assessment.

An integrated model could be used in reactor control rooms to provide operators with real-time severe accident progression to help them mitigate such an accident. The method should be quickly amenable to any complex systems analysis problem where a detailed code is used to compute potential pathways and where that code has hooks (or such can be attached) that allow for interactive querying of pathway events.

FY 2006 Accomplishments

In FY 2005 we developed and tested an MTD in single-tasking mode with a pressurizer model. In FY 2006 we developed a more general, code-diagnostic driver in C++. We used the DataCutter [1,2] framework, which provided us the infrastructure for multithreaded execution on a variety of hardware architectures, such as single and multiprocessor machines, as well homogenous and heterogeneous clusters, and distributed environments. We linked the C++ driver with MELCOR and used the driver code to modify the MELCOR input files with the help of MELCOR control functions that stop the simulations using user-specified stopping rules. The driver modifies the MELCOR input files to follow the branches and executes MELCOR with the new input files.

We developed a machine-readable format for editing rules for the input files. This file, which we call the “edit-rules” file, generates the user-input branching rules and is used by the driver to modify the input files at the stopping points to generate the new input files for the branches. The driver also updates the probabilities for the scenarios based on user-supplied branching probabilities. The driver has been registered by Sandia as the ADAPT code.

ADAPT has been successfully tested for loss of all alternating current power compounded by a loss of all feed water to the steam generators for the Zion plant for a number of key events, including failure of the surge line to the pressurizer, failure of a hot leg, containment bypass due to failure of a steam generator tube, core uncovery, the production of super-heated steam and hydrogen, and hydrogen combustion. We also developed a user-friendly, post-processing graphical capability to analyze the massive amounts of output generated by ADAPT, but the graphics package still needs some development to make it stand alone.

[1] M.D. Beynon, T. Kurc, U. Catalyurek, C. Chang, A. Sussman, and J. Saltz. “Distributed Processing of Very Large Datasets with DataCutter,” *Parallel Computing*, Vol. 27, No. 11, pp. 1457-1478, October 2001.

[2] http://www.datacutter.org

Significance

In late FY 2006, we were contacted about our work by several federal agencies – the Nuclear Regulatory Commission (NRC), DOE, the National Aeronautic and Space Administration, and the Department of Homeland Security. NRC is sufficiently interested that during a severe accident reactor safety meeting in September, they asked us to hold a workshop on the methodology we developed.

Our method could be used in the nuclear weapon, nuclear weapon transportation, waste disposal, and security areas in addition to nuclear reactor safety analysis.

Refereed Communications

A. Hakobyan, R. Denning, T. Aldemir, S. Dunagan, and D. Kunsman, "Treatment of Uncertainties in Modeling the Failure of Major RCS Components in Severe Accident Analysis," SAND 2006-0318C, in *Proceedings of the American Nuclear Society Annual Meeting*, June 2006, CD-ROM.

B. Rutt, U. Catalyurek, A. Jakobyan, K. Metzroth, T. Aldemir, R. Denning, S. Dunagan, and D. Kunsman, "Distributed Dynamic Event Tree Generation for Reliability and Risk Assessment," SAND 2006-0807C, in *Proceedings of the CLADE 2006 workshop (Challenges of Large Applications in Distributed Environments),* held in conjunction with the 15th International Symposium on High-Performance Distributed Computing, June 2006, CD-ROM.

Other Communications

K. Metzroth, U. Catalyurek, T. Aldemir, D. Kunsman, and S. Dunagan, "A Graphical Tool for the Analysis of Event Trees," SAND 2006-0319C, in *Proceedings of the American Nuclear Society Annual Meeting*, June 2006, CD-ROM.

Innovative Solar Thermochemical Water Splitting

79781

R. B. Diver Jr., J. E. Miller, R. E. Hogan Jr., M. D. Allendorf, J. N. Stuecker

Project Purpose

We investigated the use of iron oxides and mixed metal oxides containing iron (ferrites) in relatively simple, two-step thermochemical processes for splitting water into hydrogen and oxygen. These processes involve high-temperature thermal reduction of the ferrite and subsequent low-temperature hydrolysis in which the ferrite is reacted with water vapor to produce hydrogen. Thermodynamically, when the reduction and oxidation (redox) processes are combined into a cycle, it becomes a kind of "heat engine" with the output in the form of chemical energy, hydrogen.

In practice, recuperation of the sensible thermal energy in the ferrite is critical to high thermal efficiency. We have, therefore, investigated ferrite thermochemical processes, in the context of a Sandia invention, that use two sets of moving beds of ferrites in close proximity, providing radiation-driven counter-current heat recuperation. This invention also provides inherent separation of the product hydrogen and oxygen, is conceptually simple, and is an excellent match with high-concentration solar thermal technology.

Previously we established that this approach can efficiently convert thermal energy into chemical potential in the form of hydrogen. This year we experimentally and theoretically investigated various combinations of mixed-metal ferrite using a zirconia support to disperse the ferrite and improve its reactivity.

Our experimental results with cobalt-substituted ferrites mixed with yttria stabilized zirconia formed into monolithic shapes demonstrated that more hydrogen can be produced than with equivalent amounts of powders. Furthermore, we demonstrated that the mechanical and chemical stability of cobalt ferrite/zirconia monoliths can be maintained over repeated redox cycles. Our theoretical studies and experimental results suggest a new class of solid-state redox reactant and the potential for distributed, competitive, solar-thermochemical-hydrogen production.

FY 2006 Accomplishments

This year we discovered and investigated a new class of iron-based (ferrite) redox reactant material needed in the Sandia-invented counter-rotating-ring receiver/reactor/recuperator (CR5) thermochemical water-splitting heat engine. We also presented four papers on the CR5, which were published in the proceedings of the peer-reviewed ASME International Solar Energy Conference. The four papers collectively received "Best Paper Award" in the Solar Chemistry and Hydrogen Technologies track.

The priority this year was to establish whether the redox material, with the structural characteristics needed in the CR5, could be developed. To address this objective, we synthesized a variety of candidate ferrite redox materials and characterized the effect of a number of parameters on performance in a laboratory test system at the Advanced Material Laboratory (AML). We also established a solar test capability in the Sandia solar furnace and performed solar-driven water-splitting experiments. After testing a number of materials in the AML, we established a cobalt-substituted ferrite as our baseline material.

Test results of monolithic parts made with cobalt ferrite mixed with zirconia exceeded those with equivalent amounts of powders. Most importantly, structural integrity of the monolithic part was maintained even after up to 31 redox cycles. These results, along with our theoretical studies, suggest a new class of single-phase redox materials and perhaps other more effective material combinations based on cerium oxide.

We also developed a preliminary design for a proof-of-concept CR5 heat engine. The design effort was supported by computational fluid dynamic modeling. We designed the CR5 prototype to absorb approximately 9 kW of incident solar flux from the Sandia solar furnace.

As part of the CR5 prototype development, we used the Sandia-developed Robocast rapid prototyping technique to fabricate three-dimensional monolithic reactant fin sections at the actual prototype scale using alumina as a stand-in for ferrite/zirconia reactant. We also designed, developed, and fabricated the drive hardware for the CR5 prototype. Design objectives for the CR5 prototype include experimental flexibility and significant hydrogen production.

Significance

The Sandia-invented CR5 thermochemical heat engine represents a new thermodynamic construct for renewable hydrogen production. It provides an integrated design concept for driving two-step thermal reduction and water hydrolysis reactions; recuperating heat between the two reaction steps; and separating the hydrogen and oxygen product gases. However, the CR5 places severe and unique requirements on materials.

Results from our materials studies suggest the discovery of a new type of solid-state reactant in which the ferrite redox material is dissolved in or perhaps suspended in an oxygen-conducting zirconia support. The new material combinations allow the thermal reduction step to occur at higher temperatures than with the ferrite alone and to avoid detrimental slag formation. An important consequence of the materials studies is that high porosity may not be needed, and sintering may not be an issue.

In addition, other material combinations based on cerium oxide are suggested by this theory. If suitable materials can be developed and the design challenges met, the CR5 heat engine concept appears to provide an integrated approach for potentially efficient and low-cost solar hydrogen.

Refereed Communications

J.E. Miller, L.R. Evans, J.N. Stuecker, M.D. Allendorf, N.P. Siegel, and R.B. Diver, "Material Development for the CR5 Solar Thermochemical Heat Engine," in *Proceedings of the ASME International Solar Energy Conference 2006*, July 2006, CD-ROM.

M.D. Allendorf, R.B. Diver, J.E. Miller, and N.P. Siegel, "Thermodynamic Analysis of Mixed-Metal Ferrites for Hydrogen Production by Two-Step Water Splitting," in *Proceedings of the ASME International Solar Energy Conference 2006*, July 2006, CD-ROM.

R.B. Diver, J.E. Miller, M.D. Allendorf, N.P. Siegel, and R.E. Hogan, "Solar Thermochemical Water-Splitting Ferrite-Cycle Heat Engines," in *Proceedings of the ASME International Solar Energy Conference 2006*, July 2006, CD-ROM.

System Analysis of Carbon Sequestration with Clean Coal Technology

79798
P. H. Kobos

Project Purpose

The goals of this project are to characterize clean coal and carbon sequestration technologies (e.g., performance goals, associated economics); develop a research network for Sandia in the carbon sequestration research community and collaborate with other projects and within the community; and design and build a systems analysis framework and implement the framework in a system dynamics model.

The central theme of these items is to address performance and economic parameters within carbon sequestration systems. With this information, future energy systems modelers and analysts will be able to target research and development to reduce performance uncertainty and, consequently, economic uncertainty when looking to develop and potentially deploy carbon capture and sequestration technologies at the larger scale (e.g., beyond pilot projects).

FY 2006 Accomplishments

We more fully characterized carbon sequestration technologies from a systems perspective (e.g., identified performance and economic parameters). We designed, implemented, and demonstrated a system dynamics model that assesses performance and economic risk issues at both the global and local scales.

Significance

The technology characterization work, research network development efforts, and the model's design, development, and resulting analysis helped Sandia further increase its role in the carbon sequestration research community. The carbon sequestration and risk (CSR) simulation model was developed as part of this project. The model was developed in Powersim Studio, a system dynamics software package.

We developed a stronger role for Sandia within the carbon sequestration research community while also collaborating with other projects (notably the Southwest Regional Partnership (SWP) on Carbon Sequestration). The CSR model complements the DOE-sponsored SWP carbon sequestration project. Sandia plays a central role within the larger SWP team and with many outside collaborators through its ongoing development of an integrated assessment system dynamics simulation computer model.

The CSR model addresses performance and economic assessment based on risk and not on arbitrary goals (e.g., picking a number of years required for monitoring without accounting for the system's parameters based on a risk analysis), which may prove extremely useful for the broader SWP efforts. Specifically, SWP may benefit from the risk-based analytical framework and insight provided by the CSR model in the areas of assessing performance goals (e.g., sequestering carbon dioxide for a meaningful time period) and economic goals (e.g., how much might sequestration efforts cost, how might a carbon dioxide market permit trading scheme affect the system's financial viability).

With these models and the increased carbon sequestration systems analysis capabilities developed from this project, Sandia has a larger role in the national CSR community as evidenced by the project members being invited to speak at the Material Research Society and chair (and present at) carbon dioxide analysis sessions of the US Society for Energy Economics/ International Association for Energy Economics.

Other Communications

R. Klotz, P.H. Kobos, and T.E. Drennen, "Geological Carbon Sequestration: A Performance and Economic Risk Analysis," presented at the US Association for Energy Economics (AEE), International AEE Conference, Ann Arbor, MI, September 2006.

P.H. Kobos, D.J. Borns, and T.E. Drennen, "Carbon Sequestration Costs and Integrated Assessment Models: Characterizing Technology to Develop Systems Insight," presented (invited) at Materials Research Society, Boston, MA, November 2005.

MOCVD Synthesis of III-Nitride Heterostructure Nanowires for Solid-State Lighting

79800

G. T. Wang, J. R. Creighton

Project Purpose

The purpose of this project is to develop scalable processes to synthesize and characterize single-crystalline III-nitride nanowires and heterostructure nanowires for potential novel solid-state lighting (SSL) and photonic nanodevice applications.

SSL technologies, based on semiconductor light-emitting devices (LEDs), have the potential to reduce world-wide electricity consumption by more than 10 percent, which could significantly reduce US dependence on imported energy and improve energy security. The III-nitride (AlGaInN) materials system forms the foundation for white SSL and could cover a wide spectral range from the deep ultraviolet to the infrared. Single-crystalline III-nitride nanowires and heterostructure nanowires, which may possess unique optoelectronic properties, could ultimately lead to the development of novel and highly efficient SSL nanodevices.

Our approach focuses on using metal-organic chemical vapor deposition (MOCVD) to synthesize the nano-wires on 2-inch wafer substrates in a conventional cold-walled rotating disk reactor. MOCVD is a versatile, reproducible technique with excellent control that is in widespread commercial use for the growth of compound semiconductor devices, including LEDs and solid-state lasers.

We believe that success in developing MOCVD-based techniques for synthesizing III-nitride nanowires with controlled orientation and properties would represent a major advance in nanowire device research, and hence nanowire-based lighting, by allowing for the scalable and reproducible growth of heterostructure nanowires (e.g., quantum well nanowires), tunable band gap AlGaInN alloy nanowires, and n-type and p-type doped intra-nanowire junctions.

FY 2006 Accomplishments

In FY 2006 we made considerable progress in the areas of III-nitride nanowire synthesis and characterization, including obtaining a much greater understanding of the growth process and nanowire properties. By experimenting with growth on different substrate orientations of sapphire, we were able to obtain highly aligned vertical arrays of single-crystalline GaN nanowires. We grew the nanowires on r-plane sapphire by metal-catalyzed vapor-liquid-solid growth without the use of either a template or patterning. Transmission electron microscopy indicates the nanowires are single-crystalline, free of threading dislocations, and have triangular cross-sections and tip diameters of around 10 nm.

We were able to explain the high degree of vertical alignment achieved by the crystallographic match between the [1120] oriented nanowires and the r-plane sapphire surface. Using this technique, we were also, for the first time, able to grow aligned arrays of core-shell (radial) type heterostructure nanowires. Examples of core-shell type nanowires we synthesized include GaN-AlN, GaN-InN, GaN-InGaN, and the more complex GaN-InGaN-GaN three-layer heterostructure nanowires.

Using a recently developed platform at Sandia for characterizing nanowires, we probed the electrical and optical properties of the GaN nanowires. We found the optical properties of the nanowires to be strongly dependent on nanowire growth temperature, with the band-edge emission from photoluminescence experiments at around 368 nm strongly increasing in intensity as the growth temperature increased from 800 ºC to 900 ºC. The electrical properties of the nanowires also exhibited a strong dependence on growth temperature.

As the nanowire growth temperature increased from 800 ºC to 900 ºC, the resistivity decreased by approximately three orders of magnitude. We propose that at lower growth temperatures, increased carbon incorporation in the nanowires occurs, originating from the trimethylgallium precursor and leading to increased resistivity and reduced band-edge emission.

Significance

We successfully demonstrated MOCVD-based techniques for synthesizing high-quality GaN and III-nitride heterostructure nanowires for potential nanodevice applications in SSL, sensing, and nanoelectronics. Additionally, we demonstrated a method for achieving the growth of vertical, highly aligned oriented single crystalline GaN and heterostructure III-nitride nanowires on sapphire substrates without the use of patterning or a template.

Interestingly, we found that the optical and electrical properties of the nanowires are strongly dependent on the growth temperature. We postulate that this is the likely result of increased carbon incorporation in the nanowires as the growth temperature is decreased.

These advances and new understanding of the growth and properties of the nanowires will be of significant interest to the nanowire community within and outside of Sandia. The ability to use inexpensive, large-area, and process-compatible sapphire wafers for the growth of vertically aligned single crystalline GaN nanowires by MOCVD, without the use of templates or patterning, represents an important step toward realizing devices based on vertically integrated III-nitride nanowires. One such potential device would be a highly efficient LED based on dense arrays of aligned III-nitride heterostructure nanowires.

Refereed Communications

G.T. Wang, A.A. Talin, D.J. Werder, J.R. Creighton, E. Lai, and I. Arslan, "Highly Aligned, Template-Free Growth and Characterization of Vertical GaN Nanowires on Sapphire by Metal-Organic Chemical Vapor Deposition," to be published in *Nanotechnology*.

Other Communications

G.T. Wang, A.A. Talin, J.R. Creighton, D.J. Werder, and P.P. Provencio, "MOCVD Synthesis and Characterization of III-Nitride Nanowires and Heterostructure Nanowires," presented (invited) at the University of New Mexico, Albuquerque, NM, November 2005.

G.T. Wang, J.R. Creighton, A.A. Talin, and P. P. Provencio, "MOCVD Synthesis and Characterization of Aligned III Nitride Nanowire and Heterostructure Nanowire Arrays," presented at Spring Meeting of the Materials Research Society, San Francisco, CA, April 2006.

Novel System for Zero-Emission Electricity and Hydrogen Production from Coal and Biomass

79801

C. R. Shaddix, T. C. Williams, R. W. Schefer, J. C. Oefelein, A. E. Lutz

Project Purpose

This project supports efforts to develop secure, sustainable sources of electrical power and transportation fuel. In particular, the project focuses on optimizing the design of a system in which CO_2 sequestration is possible when generating both electrical power and hydrogen fuel from coal or biomass resources. To make a significant improvement in the analysis of this type of system, scientific research is being conducted in unique experimental facilities for gasification and gas turbine combustor research; detailed combustor modeling is being performed via an advanced large eddy simulation code; and a physics-based system simulation tool is being used.

FY 2006 Accomplishments

We progressed in all three major tasks: optimizing gasification for H_2 production, establishing performance parameters for a dump combustor operating on syngas fuel mixtures in oxygen diluted by CO_2, and analyzing the performance characteristics of the overall H_2 and power coproduction system.

For the gasification task, we employed an extensive set of equilibrium calculations to determine the most likely conditions for optimizing H_2 yield and gasifier throughput. We used the SKIPPY computational code to analyze the effect of particle size on gasification inhibition by the presence of H_2.

We extensively operated the Sandia dump combustor, an optically accessible 1 atm burner simulating the relevant flow and combustion characteristics of a modern stationary gas turbine. We established a set of nine canonical fuel/oxidizer mixtures, varying from a simple methane/air system (representing current operation of turbines on natural gas burning in air) to a mixture representing filtered, H_2-lean syngas burning in oxygen diluted by CO_2.

We established burner operation for a common characteristic flow condition for these different mixtures. In each case, we varied the equivalence ratio from the lean stability limit to a slightly rich stoichiometry, and measured the primary pollutants emitted from the flames. These measurements showed that combustion of syngas in oxygen diluted by CO_2 does not have adverse effects on stability limits or emissions.

We constructed a complete Simulink coproduction system model, including a simple description of the compression requirements associated with CO_2 transport and sequestration. The system modules implemented include an air separation unit, a high-temperature gasifier, gas cleanup with heat recovery, membrane separation of H_2, a gas turbine, a heat recovery steam generator, a steam turbine, a condensing heat exchanger, and a CO_2 recycle loop.

The Simulink gasifier module can be used to describe either dry feed or slurry feed of the fuel. Preliminary analysis of the effect of the gasifier equivalence ratio on system efficiencies shows that hydrogen production is optimized for an equivalence ratio of 1.7, similar to the stoichiometry at which gasifiers operate in integrated gasification combined cycle power plants.

Significance

Our accomplishments demonstrate that the notion of burning either filtered or unfiltered syngas in a gas turbine operating on CO_2-diluted oxygen should be technically feasible, assuming that suitable adjustments are made to the turbine blade design to account for the different properties of the CO_2 mixtures. Previously, no measurements had been performed on combustion in CO_2-diluted oxygen, and there was skepticism that these systems could operate near the stoichiometric limit because of excessive CO emissions.

Our work showed that this fear is unfounded. As a result, full analysis should be made of the relative energy efficiency and prospective cost of constructing and operating a coproduction system for hydrogen and electrical power based on the traditionally presumed shift reactor/hydrogen separation approach and the hydrogen separation/dilute-oxygen turbine combustion approach. This will be the focus of the system modeling performed during the final year of the project.

Refereed Communications

T.C. Williams and C.R. Shaddix, "Contamination of Carbon Monoxide with Metal Carbonyls: Implications for Combustion Research," to be published in *Combustion Science and Technology*.

Other Communications

A. Molina, C.R. Shaddix, B.S. Haynes, and F. Chejne, "Effect of Reactant Penetration on Inhibition of Coal Char Gasification," presented at 31st International Symposium on Combustion, Heidelberg, Germany, August 2006.

C.R. Shaddix, T.C. Williams, and R.W. Schefer, "Effect of Syngas Composition on Emissions from a Swirl Burner," presented at 31st International Symposium on Combustion, Heidelberg, Germany, August 2006.

T.C. Williams, C.R. Shaddix, and R.W. Schefer, "Effect of Syngas Composition on Emissions from an Idealized Gas Turbine Combustor," presented at 23rd International Pittsburgh Coal Conference, Pittsburgh, PA, September 2006.

Fuel Traps: Mapping Stability via Water Association

79803
S. L. Rempe, T. M. Alam, K. Leung, R. T. Cygan, J. A. Greathouse, J. Clawson, D. Sabo

Project Purpose

In efforts to use hydrogen and methane for energy sources and orthogonal efforts to understand and predict structure/function relationships for hydrophobic components of cell membranes, a common theme emerges regarding water as the special substance that confers stability to specific structures under specific conditions. For example, a recent study by Mao and Mao [1] shows that water forms clathrate structures around hydrogen molecules at low temperature and high pressure and releases hydrogen at high temperature, lending credibility to the new idea that water could form an environmentally attractive alternative storage compound for hydrogen fuel.

The purpose of our project is to facilitate this line of inquiry by defining the nature of interactions between hydrophobic hydrogen molecules and water by:

- determining the hydrogen occupancy of clathrate hydrates
- mapping out the conditions (T, p) for stabilizing hydrate compounds
- determining if other guest molecules enhance the stability of the molecular water framework
- defining the conditions that favor multiple occupancy of the clathrate structures by fuel molecules.

To achieve these goals, we pursued modeling studies with ab initio and classical simulation methods to map structures and stabilities of these hydrophobic compounds.

FY 2006 Accomplishments

We determined the structural and thermodynamic properties of hydrogen in liquid water and hydrogen occupation in the large and small clathrate water cages that form the clathrate hydrate crystals of interest for hydrogen storage. Specifically, we:

Completed structural studies of hydrogen cluster formation in liquid water

Radial distribution functions and hydrogen coordination numbers by water are calculated using different representations of the interatomic forces within molecular dynamics (MD), Monte Carlo, and ab initio molecular dynamics (AIMD) simulation frameworks. Although structural details differ in the radial distribution functions generated from the different force fields, all approaches agree that the average and most probable number of water molecules occupying the inner hydration sphere around hydrogen is 16. Furthermore, all results exclude the possibility of clathrate-like organization of water molecules around the hydrophobic molecular hydrogen solute, thus disputing a popular structural explanation for the peculiar properties of hydrophobic hydration.

Completed thermodynamic studies of hydrogen cluster formation in liquid water

We find that the experimental hydration-free energy of hydrogen, a slightly positive number representing the mild tendency of hydrogen to partition preferably in the gas phase, is reproduced by ab initio as well as by classical force field descriptions of hydrogen in liquid water. The challenge was to evaluate the ab initio results. We achieved this in two ways: first by evaluating the results of the AIMD simulation using the framework of the inverse quasi-chemical theory, and then by applying corrections that accounted for anharmonic fluctuations in clusters using the framework of the regular quasichemical theory.

The corrections revealed that interactions between the flexible cluster and explicit solvent water molecules account for a large fraction of the thermodynamic stability of hydrogen in water, presumably due in part to the stabilizing hydrogen bonds that occur between first and second solvation shells.

Completed structural and thermodynamic studies of hydrogen occupation in single clathrate cages
Alavi et al. [2] suggested that MD simulations obtained a single H_2 occupancy in the small cage, whereas ab initio obtained two, because the MD model allowed flexibility of the cage and included cooperative effects of the neighboring cages. Our work demonstrated that ab initio quantum chemical calculations predict the single occupancy of the small cage even without these considerations in the model if a large basis set is employed. More importantly, our work unifies ab initio quantum calculations and MD simulations, making both methods valid for determining hydrogen occupancy of the clathrate cages.

Significance

This work directly addresses our national need for complex materials that trap hydrogen. This important area of research was identified in one of President Bush's State of the Union address, and it is one with clear goals articulated by DOE.

This work raises Sandia's credibility and profile in attacking problems related to hydrogen storage and thus enhances Sandia's ability to attract outside funding from sources such as Basic Energy Sciences. It also fundamentally enhances all endeavors related to hydrophobic hydration, specifically those aimed at understanding cell processes involving the stabilization by water of hydrophobic subunits, which occurs in protein folding and other self-assembly processes.

Structural studies of hydrogen cluster formation in liquid water show classical and ab initio force fields predict nearly the same results in terms of average distribution of water density, and average and most probable numbers of water molecules surrounding hydrogen. Furthermore, both exclude the possibility of clathrate organization of water around hydrophobic hydrogen solute, thus dispelling the common structural explanation for the unique properties of hydrophobic hydration.

Our work shows that classical force fields provide a reasonable point of departure for future studies of hydrogen storage by clathrate hydrates in particular, and most likely applies to general studies of hydrophobes interacting with water and other hydrophilic media. This result can be applied to a wide variety of work at Sandia in the areas of fuel storage and biological science.

The question of what number of hydrogen molecules can be accommodated in the cages of the clathrate is significant because it determines the hydrogen storage capacity of the hydrogen clathrates. Experimental and theoretical studies of the hydrogen occupancy of the hydrogen clathrate cages have been inconclusive on this important issue. Our results agree with the latest experimental and molecular simulation works, putting this issue to rest. These same methods can be leveraged to determine the behavior of hydrogen in other complex materials investigated at Sandia.

Studies of the thermodynamic properties of hydrogen in liquid water are significant for acquiring a better microscopic understanding of how structures relate to stability in water-hydrophobic solute systems in general, and water-hydrogen systems in particular. The evaluation of hydrogen-water clusters shows that predictions of hydrogen thermodynamic properties depend critically on accounting for anharmonic thermal motions in the clusters and, more importantly, on accounting for the hydrogen bonding interactions between the cluster and the surrounding environment. This insight into the fundamental properties of hydrophobic hydration assists efforts to understand and control partitioning of gas molecules into different environments.

[1] W.L. Mao and H.-k. Mao, "Hydrogen Storage in Molecular Compounds," *Proceedings of the National Academy of Sciences of the United States of America,* vol. 101, no. 3, pp. 708-710, January 2004.
[2] S. Alavi, J.A. Ripmeester, and D.D. Klug, "Molecular-Dynamics Simulations of Binary Structure II Hydrogen and Tetrahydrofurane Clathrates," *Journal of Chemical Physics*. 124, #014704, January 2006.

The method to predict the thermodynamic properties of hydrogen in condensed phase, by first building a cluster described by an ab initio model and then correcting it, leverages work done in other projects focused on the properties of ions in complex environments. The method is generally applicable to predicting the solvation properties of any solute and thus impacts diverse Sandia investigations of ions and gas molecules relevant to biology, geophysics, water purification, and water desalination.

Refereed Communications

D. Sabo, S.L.B. Rempe, J.A. Greathouse, and M.G. Martin, "Molecular Studies of the Structural Properties of Hydrogen Gas in Bulk Water," *Molecular Simulation*, vol. 32, pp. 269-278, January 2006.

Other Communications

D. Sabo, S.L.B. Rempe, K. Leung, and M.G. Martin, "Studies of the Thermodynamic Properties of Hydrogen Gas in Bulk Water," presented at Industrial and Chemical Engineers Annual Meeting, San Francisco, CA, November 2006.

J.S. Clawson, K. Leung, R.T. Cygan, T.M. Alam, and S.L.B. Rempe, "Ab Initio Study of Hydrogen Storage in Water Clathrates," presented at American Industrial and Chemical Engineers Annual Meeting, Cincinatti, OH, October 2005.

Risk-Informed, Decision-Making Methodologies for Robust Control of Complex Infrastructures

79807

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Project Purpose

The purpose of this project is to develop, enable, understand, and model tools that will allow us to design and build robust next-generation infrastructures that can withstand both terrorist and natural threats. The premise of our work is that effective infrastructure management, be it real-time active control using a network of distributed sensors and software agents or longer-term evolutionary approaches based on policy and regulation, requires a systematic approach to identifying the significant relevant risks from potential infrastructure failures and evaluating remedial alternatives.

Current risk assessment techniques, like the N-1 contingency studies performed by electric utilities to assess the reliability of their systems, are unable to capture the growing complexity of our critical infrastructure systems that arises from their increasing scale, interconnectivity, and utilization. To address this challenge, we are developing a representative and holistic modeling framework to assess and manage risks in complex infrastructures.

Our research is being grounded through the focused study of a specific infrastructure, the bulk power grid. We formulated and are implementing a comprehensive computer model of the grid that resolves the coupled dynamics of its physical, control, and market components. This model has been integrated into a risk management framework to support improved decision making of mitigation options based upon the overall utility of decisions from the perspective of a decision maker.

We are leveraging internal expertise in infrastructure analysis and complex systems, complemented by the risk management and power systems expertise of our university collaborators, Massachusetts Institute of Technology (MIT) Professor George Apostolakis and New Mexico State University (NMSU) Professor Satish Rande.

FY 2006 Accomplishments

Our goal for FY 2006 was to implement and begin initial validation of the systems risk management framework formulated in FY 2005. Specifically, we intended to extend our bulk power model to include an improved power system simulator and a new market model layer while adapting our proposed risk management methodology to link more closely with the disruption scenarios simulated by the bulk power model.

To build confidence and refine our research direction, we sought expert opinion on our research from utilities and potential follow-on sponsors at DOE. We solicited industry feedback on our risk analysis methodology at a one-day workshop conducted with a group of staff and management from a major US utility company. Our accomplishments include the following activities:

Extended control layer of power systems model

We implemented load shedding and automatic generator control to extend the power system simulator's control layer. Another model improvement included the incorporation of a simulation scheduler, which enabled crude analyses of progressive failure (or stabilization) scenarios of the bulk grid. Recognizing limitations in current load flow simulation techniques, we initiated a new, exploratory effort in collaboration with NMSU to formulate a heuristic-based transient approximation power system model.

Prototyped market model

Leveraging existing research on "zero-intelligence agents" (ZIA), we formulated and tested a bulk power market model. In what may be the first such adaptation of ZIAs to power markets, we implemented a spot power market model with ZIA to produce qualitatively reasonable results. We first tested and verified the ZIA power market by a replica of the now-traditional ZIA model with a double auction for a hypothetical power market. We then implemented a ZIA model for a power market of bilateral trades.

Refined risk analysis methodology
We successfully adapted our MIT collaborator's infrastructure risk analysis methodology to the bulk power grid. This work included the trial application of the methodology to a partner utility, including the development of a representative value tree, constructed scales, and a consequence matrix, which were collectively used to convert the physical consequences predicted by our grid simulator into a single index, or disutility measure, that reflected the overall impact of the simulated failure scenarios to the partner utility.

Developed conceptual risk-minimization operational scheme
We made progress on the development of a conceptual agent-based management scheme that can be implemented within the framework of our power systems simulator to demonstrate improved mitigation of large-scale grid failures such as cascading blackouts. Our proposed management methodology builds on existing work by incorporating the human decision maker's value system to weigh the physical stability implications of an operational decision against the aggregated impacts of interest to the decision maker (the level of disutility) obtained by our risk analysis methodology.

Significance

This project's intermediate results show promise in addressing several pressing areas of need in grid modernization and the broader challenges associated with effective risk management of critical infrastructures. For example, mitigation of existing high-impact vulnerabilities in the bulk power grid, as required under the new North American Electric Reliability Council's critical infrastructure protection (NERC CIP) standards, requires a systematic methodology to identify critical assets whose malfunction may give rise to consequences of interest to key stakeholders and decision makers. We believe that the LDRD-developed risk analysis methodology may have near-term application to utilities for compliance with the NERC CIP standards.

The resolution of market forces is critical in any comprehensive modeling framework. Since deregulation, the practice of trading power between geographically remote utilities has become commonplace, creating large and unplanned-for stresses on the connecting grid infrastructure. Our novel model will provide an automatic mechanism for generating power orders with price dynamics that resemble a deregulated spot power market, providing potential longer-term opportunities to provide DOE with a unique modeling capability that can evaluate policy alternatives through the simulation of coupled market-grid dynamics.

Development of Design and Simulation Models for Large-Scale Hydrogen Production Plant Using Nuclear Power

80568

S. B. Rodriguez, F. Gelbard, L. A. Malczynski, R. K. Cole Jr.

Project Purpose

This project's goal is to seek a viable pathway through nuclear power to an economical, safe, and environmentally benign alternative for gasoline by using hydrogen.

High-temperature nuclear reactors can be coupled to thermochemical systems (e.g., sulfur iodine (SI)) to produce massive amounts of hydrogen. A 1,000 MW nuclear/hydrogen plant can produce as much as 100 million kilograms of hydrogen per year. Additionally, the residual heat should be sufficient to generate approximately 130 MW of electricity. For comparison purposes, one kilogram of hydrogen has the equivalent amount of energy as one gallon of gasoline. This is an area of research that is on the verge of key technological breakthroughs and thus provides Sandia a timely opportunity to become a leader in this area. Due to its cutting-edge application to large-scale hydrogen generation via nuclear reactors, an LDRD approach is highly suitable for this endeavor.

Our research indicates that when the thermochemical plant is coupled to the nuclear reactor, many system parameters will affect how much hydrogen and electricity can be produced. Thus, we are currently developing a tool, MELCOR-H2, that will simulate the entire plant in fully coupled and transient mode. Currently, no other code exists that can do all this.

We demonstrated that we can model high-temperature gas-cooled reactors (the type that would likely be used for the production of hydrogen). We added thermodynamic secondary system components such as turbines, heat exchangers, and compressors so that we can model the full secondary system. We took a modular approach so analysts and designers can fashion a secondary system according to their unique specifications – that is, the models are sufficiently generic.

We added rate kinetic equations into MELCOR-H2 so that it can also model the SI chemistry. We also added a graphical user interface (GUI) for ease of system design and analysis.

In FY 2007 we will add first-principle, advanced thermal-hydraulic turbine and compressor models, as well as an electricity generator model. We will include another well-known and well-qualified chemistry cycle, the Westinghouse hybrid sulfur cycle, and distillation and separation modules. Therefore, the models we incorporated in FY 2006, in addition to those that we will include in FY 2007, will make MELCOR the most advanced tool for hydrogen plant analysis and design.

FY 2006 Accomplishments

Our research indicated that when the thermochemical plant is coupled to a high-temperature nuclear reactor, many system parameters on the thermochemical side of the plant will affect how much hydrogen and electricity can be produced.

Our primary achievement was the completion of the first version of MELCOR-H2, which is meant to simulate the entire nuclear/hydrogen plant in fully coupled and transient mode. Currently, no other code exists that can do all this. Our research showed that we can use MELCOR-H2 to model high-temperature gas-cooled reactors (HTGRs). An HTGR is the most likely reactor type to be used to produce hydrogen.

Furthermore, we added generalized, modular thermodynamic secondary system components including turbines, heat exchangers (e.g., the intermediate heat exchanger (IHX)), and compressors that allow us to form complete secondary systems. MELCOR-H2 is not limited to a single secondary system configuration; instead, the configuration possibilities are infinite.

Additionally, we added the SI rate kinetic equations into MELCOR-H2 so that it can model the SI chemistry. This is the first time such equations were modeled successfully in transient mode. Previously, researchers completed steady state solutions. Our transient analysis approach sets us well ahead of the state of the art.

Finally, we added a GUI for ease of system design and analysis. The GUI displays system pressures, temperatures, hydrogen production, plant efficiency, and so on, and is dynamic, allowing the analysts/designers to evaluate the system as a function of time. Furthermore, the users may modify an input parameter while running the calculation and immediately observe how that parameter change affected hydrogen generation, and so on.

Significance

Energy security is an important issue for US national security and economic well-being. New concepts, ideas, and technologies are needed to improve the security and reliability of energy infrastructure and reduce dependence on imported energy. The project supports the DOE mission through the development of technology and application of this technology toward the safe and economic production of hydrogen as a carrier of energy.

Sandia is positioning itself as a world leader for the new hydrogen economy. Such economy requires the massive production of hydrogen at economical levels that only high-temperature nuclear reactors can achieve.

Because no full-scale nuclear/hydrogen system exists, we must develop models that will help us simulate such plants so that we can better design them and maximize profitability as well as plant safety. Careful economical analysis shows that the only viable way to produce massive amounts of hydrogen (say greater than 100 million kilograms per plant) on an economical basis (about $1.4 per kilogram), and without producing CO_2, can only be suitably accomplished via nuclear energy. We are now the only entity in the world that can simulate, in transient mode, a fully integrated nuclear/hydrogen plant.

Refereed Communications

S.B. Rodriguez et al., “MELCOR Extensions for Simulation of Modular Power Cycles and Thermochemical Cycles for the Generation of Hydrogen via Nuclear Reactors,” in *Proceedings of the World Hydrogen Energy Conference*, June 2006.

S.B. Rodriguez et al., “Addition of Secondary System Modules and a Graphical User Interface into MELCOR-H2—Phase 1,” in *Proceedings of the ANS Winter Conference*, November 2006.

S.B. Rodriguez et al., “MELCOR Modification for Large-Scale Hydrogen Production Using Nuclear Thermochemical Cycles,” in *Proceedings of the ANS 2005 Winter Conference*, November 2005.

S.B. Rodriguez et al., “MELCOR-H2: A Modular, Generalized Tool for the Dynamic Simulation and Design of Fully Coupled Nuclear Reactor/Hydrogen Production Plants,” in *Proceedings of the ANS Winter Conference*, November 2006.

Other Communications

S.B. Rodriguez et al., “Recent Advances in Modular Tool for the Design and Analysis of Large-Scale Hydrogen Production Using Nuclear Power,” presented at New Mexico Hydrogen Business Council, Santa Fe, NM, February 2006.

Exploiting Interfacial Water Properties for Desalination and Purification Applications

90493

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Project Purpose

Water is the critical natural resource of the new century. Significant improvements over conventional water treatment processes require novel approaches based on fundamental understanding of nanoscale and atomic interactions at interfaces between materials and aqueous media. We are examining the behavior of interfacial water at two-dimensional (2D) surfaces and in three-dimensionally (3D) confined water systems and transferring information obtained on model systems to more complex and realistic ones.

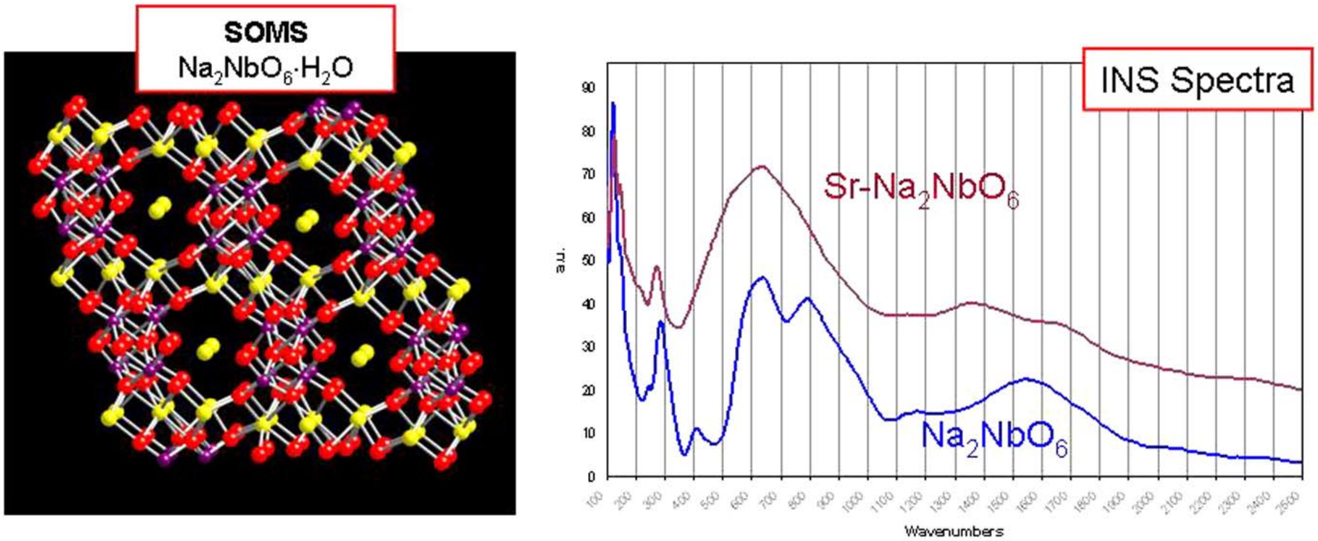
Our 2D work takes advantage of Sandia's wide ranging analytical expertise, notably in state-of-the-art microscopies and spectroscopies. We have captured glimpses into the nature of what we know to be a very complicated interfacial environment. We are examining the behavior of water on the well-defined and atomically flat mica surface as a basis for characterization and theoretical analyses. We recently extended our 2D studies to water interactions with relatively simple, self-assembled monolayers of nylon in order to investigate the nanoscale behavior of electrolytes and water at the interface of a model material more representative of commercial reverse-osmosis membranes.

Two distinct tasks comprise our 3D effort. One is using Sandia's expertise in modeling and synthesis to design and fabricate model pores of desired size, shape, and surface functionality. Primarily, we synthesize silica-based membranes with modified surface functionality designed to mimic biochannels with controlled water transport. This allows us to test concepts for improved purification in quasirealistic environments. Our second task involves using classes of zeolitic ion-exchange materials, which have properties that can be varied systematically to reveal structure-property relationships directly. In both tasks, theorists and computational chemists, working closely with the experimenters, are simulating and parameterizing the experimental results, thereby supporting data interpretation and helping in the development of new materials concepts for water treatment.

Our coordinated multidisciplinary approach emphasizes the fundamental science of interfacial water and has broad applications beyond our primary goal to direct development of materials for advanced water treatment. The influence of humidity, even in the driest of environments, and the adhesion of monolayers of water to "inert" surfaces, will influence the structure and reactivity of materials. Interfacial water will control the corrosion, stability, friction, and transport response of materials critical to Sandia's nuclear mission and other defense applications.

Understanding the fundamental behavior of these interfacial processes will ultimately help determine component reliability and stockpile survivability. Of course, this research has a role in other broad materials-based technologies, in both government and commercial sectors. Engineering and scientific efforts in electronic interconnects, microelectromechanical systems lubricants, microfluidic sensors, biosensors, fuel cell membranes, desalination, superhydrophobic materials, and other Sandia programs will benefit from a mechanism-based research effort and the development of our predictive models.

This project has broad application to several DOE programs, particularly in the Office of Science, and specifically in Basic Energy Sciences (BES). The most critical mission tie is associated with the recent congressional authorization to invest BES funding in research associated with water treatment technologies, where Sandia already is positioned to lead in a national effort due directly to the early success of this project.



SOMS zeolite structure and inelastic neutron scattering spectra indicating different water structure when Sr ion is incorporated into pore.

FY 2006 Accomplishments

We synthesized and characterized the detailed structure and chemistry of 2D polymer (nylon) surfaces and associated interfacial water. We determined that exposing nylon films to water induces a phase transition from a rigid crystalline state to an amorphous fluxional material containing roughly 8 wt% water. Interfacial force microscopy, nuclear magnetic resonance, sum frequency generation, neutron reflectivity, and other methods provided atomic-scale evidence for this behavior.

We obtained strong new evidence that water is much more viscous within nanometers of hydrophilic surfaces than in bulk. Theoretical structural modeling is under way for sodium and chloride ions in bulk ice and water, and for saltwater adjacent to muscovite, a model hydrophilic material.

Ab initio calculations and dynamic models were developed to predict thermodynamic stabilities for ions in channels, to understand how chemical groups on pore surfaces alter water structure and ion stability, and how channel diameter alters water and ion mobility across the channel. We fabricated synthetic ion channels inspired by efficient biological models and developed methodologies to control pore size, channel length, and surface chemistry with precision approaching that of natural channel systems. To couple closely with theory, we devised a platform allowing fabrication and structural and functional characterization of the synthetic pores at the individual pore level.

We compared water's role in two niobate-based zeolites. Experiments showed that water in low-exchange capacity end-member phase $Na_2Nb_2O_6 \cdot H_2O$ behaves as very rigid and immobile, more tightly bound to itself through hydrogen bonding than to cations in the zeolite channels. In contrast, water in the channel of high-exchange capacity $Na_2Nb_{1.8}Ti_{0.2}(OH)_{0.2}O_{5.6} \cdot H_2O$ behaves like fluid water, solvating the cation. Density functional modeling confirms these results. We also synthesized natural aluminosilicate zeolites to examine the mechanisms of ion exchange and hydration. Classical molecular dynamics modeling methods are being perfected to predict the energetics and local chemistry of these processes.

Significance

This project has emphasized the fundamental behavior of water at various 2D and 3D interfaces. The experimental, spectroscopic, analytical, and theoretical results provide an integrated approach to improving the design and development of new materials for waste treatment. Our 2D studies suggest that at nanometer length scales, water restructures the nylon interface, and the polymer restructures the water, both of which will have significant consequences for the transport of salts and water through polymeric membrane materials.

The restructuring generates a layering of low-density water at the polymer surface where aqueous salts are easily partitioned. A Navier-Stokes analysis in support of the interfacial force microscopic (IFM) work suggests a surface viscosity a million times greater than bulk water. This result indicates that surface functionalization may severely impede flow through membrane pores for desalination.

Molecular dynamics simulations of a spherical probe moving with respect to a planar wall also assisted in the interpretation of the IFM results. Electronic structure calculations using density functional theory for the analysis of NaCl in ice indicate the need to incorporate both ions in the model to determine the correct hydrogen bonding and hydration values. We used similar tools to examine the wetting of the basal surface of muscovite and to explain the competition between direct surface adsorption of water and the formation of a hydrogen bonded network above the surface.

Our analysis of 3D pores includes the examination of zeolite materials and the role of interfacial water in nanosized pores. SOMS (Sandia octahedral molecular sieve) is a 1D-pored molecular sieve that has high selectivity for divalent cations that are of significance in wastewater treatment. Using inelastic neutron scattering techniques and vibrational/librational signatures, we are able to study the occluded water molecules located within the pore of the channel. Inelastic neutron scattering data of the endmember SOMS show the presence of ice-like water in the pores, which was not found in the Ti-substituted SOMS.

Water is not solvating the cation but forming a rigid hydrogen-bonded network with itself and interfering with ion exchange. Nuclear magnetic resonance, density functional theory, and classical dynamics modeling and simulation studies support these findings. Similar methods are being used to examine natural zeolites (clinoptilolite) to better understand their use in ion-exchange processes for water treatment.

Another task involves the development of robust synthetic nanopore membranes engineered to achieve high water flux and high salt rejection, mimicking natural aquaporins. Combining top-down lithography and bottom-up self-assembly, we are constructing platforms that allow modification of pore size and surface chemistry, electron imaging to verify structure, and patch clamp and electrochemical measurements to determine ion-water flux.

Significant in recent findings is the successful fabrication of mesoporous membranes on 20-60 nm focused-ion-beam apertures using evaporation-induced self-assembly. This control of pore size and thickness is unprecedented and should allow fabrication of synthetic pores according to the design of natural water-ion channels, which are structured on similar length scales.

Refereed Communications

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Other Communications

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J.E. Houston, "Scanning Probe Applications to the Adhesive, Tribological, and Rheological Properties of Materials," presented at the 2006 Meeting of the American Physical Society, Baltimore, MD, March 2006.

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Development of a Universal Fuel Processor

90497

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Project Purpose

This project focuses on developing technology applicable to producing a universal fuel from diverse feedstocks readily available in North America and elsewhere (e.g., petroleum, natural gas, coal, biomass, CO_2, and water). The utility of a single logistical fuel that can be produced worldwide for all air, ground, naval, and portable power generation has already begun to be recognized within the military. Three critical research areas for the development of this technology have been identified in consultation and collaboration with contacts in the petroleum industry: feedstock transformation, fuel formulation, and fuel characterization.

During FY 2006 our efforts on feedstock transformation focused on improving the efficiency of recovering oxygen from air, one of the most costly aspects of producing synthetic fuels. We addressed soot formation, a trouble spot in the emerging autothermal reforming (ATR) technology for manufacturing synthesis gas (an intermediate in synfuel production). We also focused on selectively combining small "building block" molecules that can be derived from virtually any hydrocarbon feedstock (ethylene) into larger molecules with properties that are attractive for fuels. A high-throughput reactor system is being brought online for application to this task.

Our fuel characterization study focused on the problem of quantifying for the first time the different behaviors of two important military fuels: standard #2 diesel and JP-8 (jet fuel). This work is providing important data that can be used to validate and implement the universal fuel concept.

FY 2006 Accomplishments

Feedstock Transformation

- Constructed hybrid membrane pressure swing adsorption unit for O_2 generation
- Characterized components individually
- Modified and characterized components as a system
- Synthesized new tubular geometry O_2 selective inorganic membranes
  - O_2/N_2 selectivity = 2.3 to 3.8 depending on the coating conditions
  - Commercial selectivity = 2.1
- Conducted ATR experiments in annular laminar flow burner in rich flames at pressures up to 500 psi
  - Mapped soot formation as function of pressure and reaction stoichiometry
  - H_2 production is sensitive to flow velocity, oxidation stoichiometry, steam content, and mixing configuration
  - CO_2 participates in the burner chemistry, but does not appear to increase soot formation

Fuel Formulation

- Oligomerized ethylene into fuel range molecules
- Succeeded in activating isobutane in an oligomerization environment
- Acquired and began assembling high-throughput experimental apparatus

Fuel Characterization

- For the first time, we demonstrated that JP8, when used in diesel fuel injectors, exhibits significant differences compared to standard #2 diesel fuel. These differences include:
  - Faster evaporation and shorter liquid spray penetration
  - Longer ignition delays
  - Faster heat-release rates at the time of ignition.

Significance

The development of technologies to produce liquid transportation fuels from diverse feedstocks will support the DOE strategic energy and environment goals and the goals of Sandia's Energy, Security, and Defense Technologies Division. This research enhances Sandia competencies including combustion research and materials development. The application of the new high-throughput reactors to this problem will allow new and unexplored chemistries to be developed and eventually exploited. In particular, reactions of the CO_2 content of syngas or recovered from other sources are being targeted.

The feedstock transformation results provide information and technology for improving the energy efficiency, size, and capital costs associated with oxygen generation and provide important insights into the robustness and operability of ATR reactors.

The fuel characterization task has for the first time provided quantitative comparisons of the in-cylinder behavior of two important fuels, and the data is already attracting the attention of fuel producers and end-users who will use the data to help validate the universal fuel concept and to determine the necessary modifications to more fully implement the concept.

Rapid Updating of Stochastic Models Using Sensor Information

90501
S. A. McKenna, E. D. Vugrin, K. A. Klise

Project Purpose

This project with the University of Texas at Austin (UT) is focused on developing techniques for rapid updating of models conditional to real-time sensor data where the model properties themselves are uncertain. Achievement of this goal will enable these techniques to have widespread application in many fields where real-time data streams must be tied to complex numerical models for real-time prediction.

We identified approaches developed in electrical engineering and signal processing fields, specifically the ensemble Kalman filter (EnKF), and updated them to work in areas of earth sciences with focused applications in petroleum and water systems. As background, in the EnKF scheme, a suitable number of initial stochastic representations of the state variables (model parameters) are sampled from appropriate probability distributions to construct a state matrix.

This state matrix is updated gradually and sequentially using real-time sensor-based observations of a system output until the predicted output variables of the system (e.g., petroleum production rate, and groundwater flow) converge with the measured values. A covariance matrix between the state and the output variables is determined considering the complete set of stochastic representations (ensemble). The covariance between the state and output variables is normalized to obtain an adjustment factor for each state variable, called the Kalman gain, that is used to update the initial state matrix. The updated state matrix represents the updated distribution of uncertainty in the predicted variable with respect to the set of uncertain model parameters.

Work has focused on four potential applications:

1. Prediction of secondary oil recovery (water flood) conditioned to well data and dynamic injection and production rates
2. Estimation of mass-transfer rates between solute and pipe-wall material in a water distribution network
3. Conditioning of a physical-process-based depositional model to data observed in wells
4. Constrained extraction of groundwater from an aquifer.

This work has developed approaches for the rapid updating of model states as sensor data become available.

FY 2006 Accomplishments

Accomplishments over the past 12 months are focused on further refinement and application of EnKF techniques, development of instrumented experimental facilities for generating real-time data streams, and development of approaches for real-time control of systems.

We constructed two laboratory-scale experimental facilities:

1. to implement a real-time control algorithm for optimizing oil production in the presence of water coning. We performed a series of experiments with sand beds that exhibited varying patterns of heterogeneity and compared the observed responses against results from numerical simulation and analytical models.
2. to optimal-control well performance during a gas flood in an oil reservoir. This experimental setup was designed and developed to mimic gas injection processes and to gather data that can be used within an optimal control scheme to maximize oil recovery. We also performed experiments with variable injection rates, size of the initial gas cap, vertical wells, horizontal wells, and different combinations of open well completions.

Both experimental setups are complete with an automated data acquisition system, programmable control valves, and a dedicated online computer.

We developed numerical techniques for optimal control of the gas injection process using response surface and nonlinear sequential programming. We also implemented a procedure for calibrating a response surface that characterizes the relationship between the reservoir response and the operating parameters. The response surface is used as a proxy model within the sequential optimization procedure to arrive at a set of optimal operating parameters (e.g., gas production).

We applied the EnKF technique to the optimal updating of an aquifer model of the Barton Springs aquifer in Texas. This application uses daily water level measurements and pumping rates from wells in the aquifer to predict the daily spring flow at Barton Springs. The EnKF approach succeeded in updating the hydraulic conductivity model of the aquifer where other, more traditional, parameter estimation techniques failed. Using a simplified Barton Springs aquifer model, we accomplished a second approach to parameter estimation.

We conditioned the sedimentary process models to well data using EnKF. This included extension of the one-dimensional sedimentary model to account for variable bed slopes, implementation of principal components to control the erratic convergence observed when the iterative updating was applied, and development of approaches for taking into consideration the uncertainty in several state parameters (sediment velocity, grain size distribution, and so on).

Significance

The science and technology significance of this project is that EnKF techniques developed for very different applications can be extended and applied to earth science and infrastructure problems. These applications are generating considerable interest from the petroleum industry where the installation of "smart well" technology is generating large amounts of real-time data that are currently underused. Approaches developed in this project can be employed to use those data streams to reduce uncertainty in reservoir properties and control secondary recovery processes. We are demonstrating that the same approaches are applicable to the development and production of groundwater resources; however, for the most part, the groundwater industry is not yet at the same level of interest in real-time control of resources as is the petroleum industry. We expect that interest from water utilities and groundwater management districts will increase as resources become increasingly developed. This work positions Sandia and UT to be on the forefront of this increasing focus on water resources.

The experimental setup and the plan of experiments are unique in that previous research in this area has all been theoretical, with little effort spent on evaluating the efficacy of the proposed algorithms. The design of the setup permits conducting experiments with various configurations and mimics flow and well control in a real field setting, thereby ensuring that the experimental results would be relevant in a field setting. Both experimental setups address common real-world problems experienced in the petroleum industry (water coning and gas injection).

Other Communications

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Analysis of Real-Time Reservoir Monitoring: Reservoirs, Strategies, and Modeling

90729

S. P. Cooper, B. E. Jakaboski, M. J. Rightley, C. J. Weiss, S. S. Mani, B. G. van Bloemen Waanders, R. A. Normann, J. C. Lorenz

Project Purpose

The primary goal of this effort was to prove the feasibility of linked reservoir monitoring and modeling to encourage the use of future instrumented wells that use down-hole sensing and flow control to increase production. Such instrumented wells, known as "smart wells," are being successfully used in the North Sea and the Middle East in high-rate wells to identify and isolate sections of wells in advance of water entry.

This technology is used much less frequently in US reservoirs in part because of the expense, but also because the advantages of their use to overall increased production are less clear in mature fields. This project provides that clarity by providing explicit estimates of the economic value of such technology, expanding the modeling and control underpinning for a system that incorporates the sensor data into both refined reservoir models and improved real-time production control, and examining the feasibility of long-term (> 5 year) survival of such sensors in the hostile borehole environment.

FY 2006 Accomplishments

This project consisted of three parts: a value of information (VOI) analysis to address the economic advantages, reservoir simulation modeling and control optimization to prove the capability, and evaluation of new-generation sensor packaging to survive the borehole environment for long periods of time. The first two were joint efforts between the University of Texas at Austin (UT) and Sandia; the last was conducted entirely at Sandia.

We made progress on the VOI decision tree analysis of technologies, sensors, and reservoirs. VOI scenarios, with a high ranking for increased oil and gas ultimate recovery using new technologies, developed in this project include: mature light oil fields (Permian Basin), deep water offshore field (Gemini), tight and shale gas-unconventional fields (Mesaverde), and heavy oil-steam flood candidates (Kern River). Each is a future candidate for industry partnerships and pilot studies.

We developed initial codes in order to integrate data from several of the proposed sensors into reservoir models. These have shown that the incorporation of continuous data from pressure, saturation, and flow velocity sensors has a significant impact on the reservoir models to predict known permeability parameters within the reservoir.

Finally, we conducted sensor packaging analysis by defining the parameters of the borehole environments in which the sensors must survive for long periods (minimum of five years) of time. These parameters included such things as temperature, pressure, and fluid chemistry.

Significance

This project was designed to enhance our nation's energy surety and security interests through the development of advanced modeling techniques and next-generation sensors to enhance domestic production.

This work has been a successful and productive joint effort that has generated numerous potential future opportunities for collaboration between Sandia and UT's Bureau of Economic Geology, Department of Petroleum and Geosystems Engineering, and the Institute of Computational and Engineering Mathematics.

Other Communications

R. Banchs, H. Klie, A. Rodríguez, and M.F. Wheeler, “A Neural Stochastic Optimization Framework for Oil Parameter Estimation,” in *Proceedings of the International Conference on Intelligent Data Engineering and Automated Learning (IDEAL)*, September 2006, CD-ROM.

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Merging Spatially Variant Physical Process Models under an Optimized Systems Dynamics Framework

90730
T. S. Lowry, V. C. Tidwell

Project Purpose

The purpose of this research is to create a coupled integrated modular simulation framework (IMSF) rapid dispute prevention process by integrating spatially detailed water models, systems dynamics models, and stakeholder negotiation processes. The approach will create two "bridges:" one that spans the gap between data, models, and decision analysis, and another that spans the gap between information, understanding, and decision making. Each bridge enables two-way communication between the connected parts to allow the system to be used in an iterative manner, both internally and externally, for model optimization and collaborative negotiations.

Several scientific advances are necessary for the success of this project:

- The development of a methodology by which spatially distributed models, systems dynamics models, and decision analysis models can communicate with each other and draw from the same set of data (data to models).
- The creation of an optimization process whereby each model can be automatically calibrated to a user-defined set of metrics (model to model and model to decision model).
- The development of an intelligence-gathering process that scientifically describes and uses stakeholder input (data to models).
- The design and construction of a decision optimization methodology that promotes formation of management alternatives based upon the latest quantitative hydrogeological data and operational constraints that are amenable to all parties involved (information to understanding to decision making).

This research is a collaborative effort between Sandia and the University of Texas at Austin (UT). The Barton Springs segment of the Edwards Aquifer in Austin, TX, is the test bed for this project because of its rich data sets, the availability of existing groundwater flow and transport models, and an active stakeholder process.

FY 2006 Accomplishments

In FY 2006 this project centered on software development, research and adoption of automated optimization processes, refinement of the current physical process and systems dynamics models, development of new and scientifically based methods for gathering and using stakeholder intelligence and information, and formation of procedure-based policy development methods. Specifically we:

1. Built a functional IMSF that bridges the gap between systems dynamics models and physical process models. The IMSF incorporates a graphical user interface to allow a user to populate the appropriate input files and run either a Powersim-based systems dynamics model or a MODFLOW groundwater flow mode.
2. Included the ability to use multiobjective optimization to examine the system for solutions that fit user-defined metrics. The optimization is based on a metaheuristic tabu search algorithm and is incorporated as part of the IMSF.
3. Used the IMSF as part of a series of consensus-building exercises for and developed a stakeholder process whereby the IMSF can be used to reach group consensus on multiattribute systems.

Significance

The IMSF has been developed in a generic manner that allows it to be used and applied to develop resource management alternatives in other settings. Already, the IMSF has been slated for use in a 2007 LDRD project that will examine the water-energy nexus. In addition, a strategic partnership was formed between the UT-Sandia team and the Texas Water Development Board to evaluate the potential use and implementation of the IMSF approach to regional-scale water allocation problems in Texas. This has led to a class formed at UT that examines the connection between science and policy and uses the IMSF in its modeling work.

The IMSF is also being evaluated for use on the Willamette River in Oregon, where it could form the backbone of a modeling system used to establish and evaluate a temperature credit market system. The capabilities extended through the development of the IMSF may be used in other systems analysis work. This gives a great advantage to Sandia for addressing resource-based issues.

Refereed Communications

M. Cornelius, S.A. Pierce, and J.M. Sharp Jr., "Spatial Analysis of Urban Infrastructure Density as a Function of Land Use for Refining Aquifer Recharge Estimates," in *Proceedings of the NGWA Groundwater Summit*, April 2006, CD-ROM.

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S.A. Pierce, J.M. Sharp Jr., T.S. Lowry, and V.C. Tidwell, "Decision Support Theory and Sustainable Management of the Karstic Edwards Aquifer," presented at the Geological Society of America Annual Conference, Salt Lake City, UT, October 2005.

Energy Infrastructure Surety for Military Applications

93552

A. A. Akhil, J. W. Stevens III, L. R. Phillips, J. W. Ginn, T. F. Corbet Jr.

Project Purpose

The purpose of this project is to develop a set of tools that enables a military base to install an energy surety microgrid that is resistant to the kinds of vulnerabilities that a "normal" electric utility infrastructure is subject to when it is stressed by a malicious attack, a natural disaster, or extreme fuel supply restrictions.

A key tool is the development of a consequence model that relates the reliability provided by the microgrid to the core mission of the base and the ability to accomplish that mission under a natural or man-made threat condition. Other tools include the methodology to optimize the energy storage (electric, fuel, and thermal) within the energy surety microgrid.

It is anticipated that the first use by the military at key bases will provide a sufficient learning process such that a refined, tested methodology can then be applied to civilian applications.

FY 2006 Accomplishments

1. In collaboration with the Army Construction Engineering Research Laboratory (CERL), we conducted a base selection process. Based on a match of technical criteria with project goals, Ft. Sill was selected as the candidate base.
2. A first iteration of the consequence model designed to assess infrastructure impacts at a US Army Training Base was completed and reviewed in collaboration with CERL. Further refinements to the model will follow with specific information from the base.
3. New Mexico State University completed the first iteration of the energy storage methodology. Further refinements will continue during Phase II.
4. We documented the fundamentals of the energy surety concept in the areas of storage and controls for the energy surety microgrid.

Significance

We published journal articles in *Public Works Digest* and the *Distributed Energy Journal*. These articles and other publicity sparked interest in the energy surety microgrid by the other branches of service, and we received inquiries from the Air Force and the Navy.

This project has attracted a great deal of outside attention because it integrates various elements of distributed generation, energy storage, and controls to create a microgrid on a military base. The related consequence model characterizes the microgrid performance in terms of mission readiness/capability that are clearly understandable by a military base commander. Similarly, the methodology being developed for optimization of energy and fuel storage is a new approach that has not been addressed in any prior research. It leverages Sandia's existing capability in energy storage to develop a methodology that is applicable beyond surety microgrid needs

Hybrid Inorganic-Organic Polymer Composites for Improved Performance in Polymer-Electrolyte Fuel Cells

93554

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Project Purpose

The importance of improved fuel cell development is evidenced by the *National Hydrogen Energy Roadmap* issued by the DOE in 2002 that articulated the needs and requirements of a hydrogen economy. A hydrogen economy represents an environmentally clean alternative to foreign oil, improving the security and surety of our energy needs. In order for it to be successful, many technical hurdles must be overcome.

One important ancillary technology that has received attention is polymer electrolyte membrane (PEM) fuel cells. PEM fuel cells are the leading candidates for battery replacement in portable electronics and energy converters for automotive and stationary applications. A primary technical deficiency of PEMs is poor performance at low relative humidities because of poor proton transport in the polymeric membrane. This limitation complicates fuel cell systems and balance of plant design, since the membranes must be near 100 percent humidified at all times.

Because of these humidity limitations, fuel cells using organic polymer membranes can operate only at temperatures < 100 ºC. On the other hand, many inorganic solids conduct protons efficiently at high temperatures under dry conditions; however, these materials form membranes with poor physical properties (brittle, water soluble). The development of polymer electrolytes that can operate between 120-150 ºC and at lower relative humidities will result in more efficient fuel cell systems that function with higher energy conversion and simplified cooling systems.

We propose to develop the next generation of organic/inorganic hybrid proton conducting membranes that have a well-defined nanostructure where the inorganic component is attached directly (through a chemical bond) to the inorganic phase. Organic/inorganic composites are ideal materials for next-generation PEMs because they combine the facile processability of polymers with the high temperature stability, functionality, and reinforcement attributes of inorganic materials.

Simply blending inorganic particles and polymers does not produce advantageous properties, and water-soluble inorganic acids often tend to “leach out” when physically incorporated into the polymer by mixing. Our approach addresses this issue by creating intimate contact between the polymer chains and inorganic nanoparticles, which will promote well-defined proton conduction pathways. This will result in highlyconducting materials that can meet the goals of conductivity at low relative humidity and mechanical stability at high temperatures.

This project is not without risks, as hybrid polymer composites consisting of proton conducting inorganic acids, which are chemically bound to the organic polymer, have not been extensively studied for fuel cell applications. Thus, it is not known how the blending of these two moieties will affect their individual properties. Successful completion of this project will lead to improved fuel cell function that can be leveraged by Sandia and other governmental entities including DOE, the Department of Defense, and the Office of Naval Research.

FY 2006 Accomplishments

The project consists of two main research areas: the physical incorporation of heteropolyacids (HPAs) into the sulfonated Diels-Alder poly(phenylene) (SDAPP) polymer, and investigation into the synthesis and characterization of inorganic silica nanoparticles to act as templating agents for ion-containing block copolymers.

We screened known HPAs to determine the most promising candidates for enhancement of proton conductivity. To narrow down potential candidates, we are testing their compatibility with the SDAPP membrane by incorporating them into the polymer using physical methods and testing their conductivity. We prepared composite HPA-SDAPP membranes via two methods. In the first, we dissolved HPA and the salt form (Na^+ counterion) of SDAPP individually in dimethylacetamide solvent, then mixed the solutions together, stirred for several hours, then cast and dried. We protonated the hybrid films by boiling them in 0.75 M H_2SO_4 for 45 minutes and soaking them in boiling H_2O for an hour. Given the water-solubility of HPA, it is possible that some HPA leached out of the film during the protonation step, as evidenced by a slight color change of the acid solution and/or the film for some samples. Thus, we can't be assured that the amount of HPA originally incorporated into the film remains in the converted sample.

To avoid this leaching problem during the acidification step, we attempted another method of HPA impregnation: imbibing. In this method, we soaked already-protonated SDAPP films (H^+ form) in solutions of varying concentrations of PMA in H_2O for 48 hours at room temperature. We measured ionic (proton) conductivity of fully hydrated membranes in water by four-probe electrochemical impedance spectroscopy. Proton conductivity provides a relatively quick and easy screening method to determine whether a candidate has enough promise to pursue as a hybrid polymer. We saw up to a 13 percent increase in conductivity of the HPA-SDAPP hybrids over the pure SDAPP polymer. Analysis and characterization of the physical and chemical structure of these films is under way.

Another approach to the creation of hybrid polymer composite membranes entails building a functionalized inorganic matrix (silica) and growing the polymer around it. Initially, we used atom transfer radical polymerization to try to grow a sulfonated proton-conducting polymer off the surface of the silica nanoparticle, but attempts to polymerize p-styrene sulfonate off the silica in water/methanol were unsuccessful.

We are investigating alternative polymerization strategies to resolve molecular weight issues, including direct attachment of an already-synthesized polymer to the silica surface. Other strategies we are pursuing include directly attaching sulfonated groups to the silica nanoparticle surface and investigating sulfonated bridged polysilsesquioxanes for use as the inorganic component of the hybrid composite membrane.

Significance

One goal of the DOE's Hydrogen, Fuel Cell, and Infrastructure (HFCIT) Program is to reduce fuel cell system cost and size and improve the performance and durability of fuel cell systems for transportation, and for small, stationary and portable applications. Significant emphasis is being placed on development of advanced polymer electrolyte membranes that can operate at higher temperatures and lower relative humidities.

One goal of Sandia's Energy and Infrastructure Assurance (EIA) is to enhance the surety and sustainability of the energy infrastructure, including developing next-generation energy technologies. Development of advanced fuel cell membranes will help fulfill Sandia's mission to "ensure clean, abundant, and affordable energy" and thereby strengthen national energy security. It will also promote EIA's mission to sustain a core of science that supports advanced energy technologies and programs.

This project will benefit the EIA energy surety investment area by advancing the state of the art of PEM fuel cells, which will in turn advance the US hydrogen economy. Fuel cells that operate at higher temperatures will find applications in automotive and portable power. Fundamental studies of the structure-property relationships of these membranes will advance the general knowledge in this relatively new research area and may lead to programmatic funding from entities such as the DOE HFCIT Program, and possibly direct industry funding.

Sandia is an emerging player in the DOE fuel cell program, with strong capabilities that have resulted from LDRD investment. Successful completion of this project will lead to improved fuel cell function that can be leveraged by Sandia's Military Technologies and Assessments and Nonproliferation and Assessments group for defense and national security applications.

Enhanced Biomass to Bioenergy Interconversion through Protein and Metabolic Engineering

93555

B. A. Simmons, J. V. Volponi, G. M. Buffleben, R. Sapra, D. C. Roe, J. M. Faulon

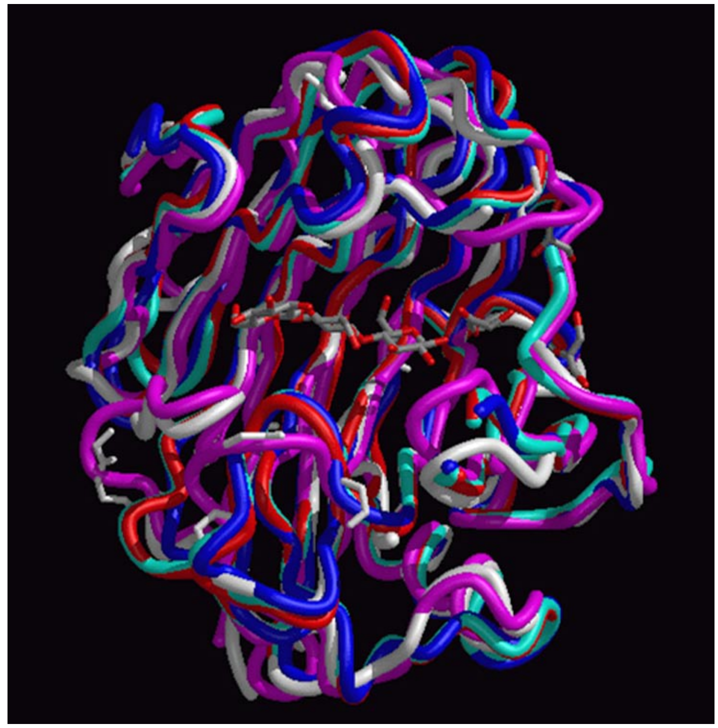
Project Purpose

The dependence of the United States on imported foreign energy supplies, specifically petroleum and petroleum-based derivatives, has become a growing national security issue. It is estimated that over the next 20 years, energy consumption in the US will rise by 30 percent, but domestic energy production will grow by only 25 percent. Imports of petroleum currently supply more than 55 percent of domestic needs; that need is expected to grow to more than 68 percent by 2025 as global demand continues to rise and domestic oil production continues to decline.

The transportation sector depends almost entirely on petroleum fuels (97 percent) to meet current energy demands and accounts for the consumption of approximately 200 billion gallons per year of nonrenewable petroleum-based (gasoline and diesel) products. In this context, it is important to find a national approach to transportation fuel generation that produces a diverse range of feedstock options to create a robust, economical, and efficient system of availability and distribution.

There are several approaches capable of producing biofuels. One attractive approach is to convert biomass (agricultural, forestry and industrial wastes, municipal solid wastes, and crops grown solely for energy purposes) into ethanol. A significant amount of research has been conducted over the past four decades to make this process cost effective and reasonably simple to operate, although significant obstacles still remain.

While corn feed is currently the main source of ethanol production, the largest available feedstock for this process lies in the form of cellulosic and lignocellulosic materials. Currently, no viable platform of refining cellulosic material into ethanol exists, and one of the primary needs is improved enzymes. We are attempting to produce robust and highly active enzymes that can significantly enhance the conversion of cellulosic material into ethanol.



Structural alignment showing backbone of set of homologous endocellulase crystal structures. Substrate shown in gray with red OH groups.

FY 2006 Accomplishments

We have met or exceeded all FY 2006 milestones. In year one, we focused primarily on improving enzymes for the hydrolysis of cellulose. Specific characteristics required for the enzyme to be active for the pretreatment of the cellulosic raw material include thermotolerance and pH stability. In combining pretreatment with enzymatic hydrolysis, the desired range of operations spans from 60-80 ºC and from pH 2-4. However, instead of engineering pH and temperature tolerance into the enzyme, we chose to work with enzymes from extremophilic archaea. We developed computational tools capable of predicting the impact of mutations on both the kinetics and thermostability of cellulase enzymes.

We focused on the sso1949 gene, which encodes a cellulase enzyme that is thermostable and exhibits an exceptional activity at extremely low pH. This combination of acid and heat stability has not yet been reported for any other cellulase. Our objective is to engineer this enzyme to be a highly efficient catalyst as defined by high turnover and low binding affinity. This will be accomplished by implementing the changes in the genetic sequence of the enzyme as dictated by the computational effort.

We obtained a clone from the laboratory of Dr. G. Lipps at the University of Bayreuth in Germany and successfully expressed this protein using recombinant expression systems. Our next step will be to characterize the baseline kinetics of the protein as compared to other enzymes, and then modify the DNA sequence to produce mutants that will then be screened for the desired characteristics.

Significance

Ethanol production from biomass is a stated goal of the DOE, and, the Energy Policy Act of 2005 mandates a three-fold increase in the amount of ethanol to be mixed with gasoline sold in the US to triple the current capacity (7.5 billion gallons) by 2012. With this precedent, as well as recent developments in the realm of biofuels, bioethanol is the clear priority for near-term biofuel production in the US.

The development of robust enzymes that can enable the realization of consolidated bioprocessing of ethanol production would be a significant advance in the field of biofuels. The enzyme engineering techniques used at Sandia, combined with the enhanced performance aspects of extremophiles, will be one of the first significant steps toward the realization of an integrated pipeline of cellulosic ethanol production.

Refereed Communications

R. Sapra, D. Roe, J.L. Faulon, G. Buffleben, J. Volponi, and B. Simmons, "Enzyme Engineering of SSO 1949 for Enhanced Cellulase Activity," in *Proceedings of the International Conference on Metabolic Engineering*, 2006.

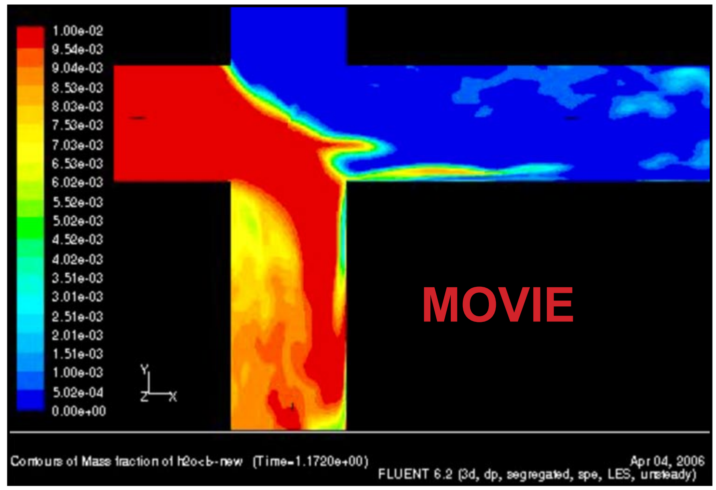
Joint Physical and Numerical Modeling of Water Distribution Networks

93556

S. A. McKenna, J. L. Wright, S. W. Webb, T. J. O'Hern, C. K. Ho, B. G. van Bloemen Waanders

Project Purpose

Threats to water distribution systems from the accidental or intentional release of contaminants are currently poorly understood due in large part to a lack of understanding of how these contaminants move through the distribution system. A better understanding, and validated computational models, of the flow in water distribution systems would enable determination of sensor placement in real water distribution networks, allow source identification, and guide mitigation/minimization efforts.



2D view of a 3D simulation of turbulent mixing in a cross joint created using the large eddy simulation approach at a Reynolds number of 40,000. Note the transient shedding events at the fluid interface.

The overall goal of this project is to accurately determine the solute mixing processes within water distribution networks through closely linked laboratory and numerical work at increasing levels of scale. The current state-of-the-practice network simulators assume solute mixing is complete at each network junction, and solute then follows the flow distribution in the pipes leading away from the junction. Our work shows that this idealized behavior can be quite far from the actual mixing behavior.

The two major components of this research are:

1. Experimental determination of the amount of solute mixing in pipe networks, both at a single joint and across a scale network consisting of multiple joints linked together
2. Numerical modeling to both elucidate the physical processes responsible for mixing and provide a basis for extrapolating physical processes captured in high-resolution models up to coarser scales that can be modeled with less computationally intensive techniques.

We divided the work into four tasks: experimental, microscale (single joints of pipes and the related high-fidelity process modeling), mesoscale (transfer of mixing processes from single joint to multiple joints in a portion of the network using less-resolved models), and macroscale (the experiments and modeling done at the scale of the entire network) modeling.

The basis of the experimental work is the tracking of a salt solution through the single joints and the network using conductivity meters. The experiments have in-line flow and conductivity meters connected to data logging systems to provide real-time acquisition and display of the experimental data. These meters produce instantaneous measurements of flow and conductivity at 0.5-second intervals. The experimental facility constructed for this work can serve as a "user facility" to other organizations, including water utilities, academia, and research organizations in the areas of distribution networks and SCADA (supervisory control and data acquisition) systems.

We employed two commercial computational fluid dynamics (CFD) codes to identify the mixing processes: FLUENT models fully transient turbulent flow within single joints (microscale) using a large eddy simulation (LES) formulation, and CFD models steady mixing at the same scale as well as in a series of joints (mesoscale).

FY 2006 Accomplishments

We had accomplishments in both the experimental and numerical modeling aspects of this project.

We completed more than 1000 experiments of mixing in single-joint junctions. We performed these experiments on three different junction geometries: cross, double-T, and U. For each geometry, we examined a range of Reynolds numbers for input and output flows. For the cross joints, we varied the pipe diameter between 0.5 and 2 inches. This testing is still under way and has presented various challenges in obtaining accurate measurement and reliable data summary and presentation strategies.

We developed a high-fidelity CFD model using the LES approach to understand the physical processes responsible for mixing in crosses and double-T joints. These simulations identified transient instabilities at the interface between the ambient and tracer fluids within the pipe joint as the source of the mixing.

We developed CFD models of mixing in single joints (cross joints and double-T joints) using the two-equation (k-epsilon) turbulence model to solve the turbulent kinetic-energy and energy-dissipation equations along with the continuity and turbulent momentum equations. We demonstrated, via comparison with experiments, that the turbulent Schmidt number (and, hence, turbulent diffusivity) can be used as a scaling parameter to model the enhanced mixing caused by transient instabilities at the impinging interface within the junction.

We developed CFD models of mixing in multijoint networks and showed that the predicted mixing behavior matched well with experimental data along various sections of the network when the appropriate turbulent Schmidt number was used.

Significance

Through this work, we have gained a number of significant technical insights regarding fundamental aspects of mixing in pipe joints. Specifically, the experimental work has shown that:

- Mixing ratios in a single joint can be as large as 90/10 for cross junctions for equal input and output flows.
- Mixing ratios are essentially independent of pipe diameter.
- Mixing ratios are only slightly dependent on Reynolds numbers, with decreased mixing (increased ratio) occurring with increasing Reynolds numbers.
- Of all factors considered, the separation distance between the T's and the limbs of a U joint is the dominant factor in determining the amount of solute mixing with larger separation distances (i.e., 20-40 pipe diameters), producing mixing ratios that are almost 50/50 for equal input and output flows.

To date, computational models have demonstrated that mixing in a single pipe joint is controlled by small-scale, transient shedding events along the interface between the tracer-ambient fluids. The bulk effect on solute mixing of these events can be modeled effectively at coarser scales by using the Schmidt number as a scaling parameter. These results demonstrate that it should be possible to capture the salient features of turbulent mixing seen in the experiments and in the highly resolved CFD models in less computationally intensive network simulation tools.

The experimental work conducted in this project shows that the international industry standard software for modeling solute transport within distribution systems is incorrect. This inaccuracy has been documented at the scale of a single joint, but the full impact of this inaccuracy across a large network model with many joints is still being determined.

We suspect that the impact of this work over the long term will be to change the fundamental approach used in network models and improve the ability of these models to predict the movement of contaminants within distribution systems.

This work has made a positive addition to Sandia's already strong reputation in the area of water distribution network security. Other efforts in the distribution network security area at Sandia include the determination of optimal locations for locating sensors within networks and the use of data from these sensors for inverse localization of the contaminant source. Both of these technologies depend strongly on an accurate model of solute transport within the respective network. Work in this project shows that the current models underlying those technologies are inaccurate, and we are developing techniques for improving them.

Other Communications

P. Romero-Gomez, C.Y. Choi, B. van Bloemen Waanders, and S.A. McKenna, "Transport Phenomena at Intersections of Pressurized Pipe Systems," in *Proceedings of the 8th Annual Water Distribution System Analysis Symposium, (WDSA06)*, August 2006, CD-ROM.

C.K. Ho, L. Orear. Jr., J.L. Wright, and S.A. McKenna, "Contaminant Mixing at Pipe Joints: Comparison Between Laboratory Flow Experiments and Computational Fluid Dynamics Models," in *Proceedings of the 8th Annual Water Distribution System Analysis Symposium, (WDSA06)*, August 2006, CD-ROM.

S.W. Webb and B. van Bloemen Waanders, "High-Fidelity Computational Fluid Dynamics for Water Distribution Systems," in *Proceedings of the 8th Annual Water Distribution System Analysis Symposium,* August 2006, CD-ROM.

S.W. Webb and B. van Bloemen Waanders, "Validation of Mixing in Crosses and Tees Using the LES Method," presented at Fluent Users' Group Meeting, Monterey, CA, May 2006.

Computational and Experimental Study of Nanoporous Membranes for Water Desalination and Decontamination

93558

B. J. Debusschere, E. Cruz, M. S. Kent, H. Adalsteinsson, B. A. Simmons, R. V. Davalos, H. N. Najm, K. R. Long, P. Ponce, E. S. Lee, M. A. Hickner, Z. J. Chavez

Project Purpose

Because of the ever-growing demand worldwide for high-quality drinking water, the ability to exploit new water sources and efficiently reuse existing water supplies is essential. In this context, the development of improved water purification and desalination techniques plays a very important role.

Industry standard purification and desalination techniques mainly use reverse osmosis filtration (RO) or electrodialysis (ED). The operational efficiency of these processes is limited by the membranes used. The design of improved membranes, however, is hampered by a lack of fundamental understanding of these filtration processes on the nanometer scale of the membrane pores.

The purpose of this project is to investigate and develop a working knowledge of both the relationship between membrane properties and operating conditions and the membrane contaminant rejection (or selectivity) characteristics and susceptibility to biofouling. To achieve this goal, we are employing a combination of multiscale modeling and experimental design with correlated validation between the two, and are developing multiscale models that employ molecular and Brownian dynamics coupled to continuum mechanics. We will use these validated models to suggest directions for further improvement in the membranes used for RO and ED. The project targets membranes consisting of well-defined nanopore arrays, phase-separated polymers, and conjugated polymers.

Since modern synthetic techniques allow ever more control to be exercised over the physical characteristics and porosities of the membranes on the nanoscale, the detailed understanding gained from this study will enable the science-based optimization of the membrane structure in order to achieve drastically improved filtration and desalination performance.

FY 2006 Accomplishments

We made progress on all three fronts of this project:

- Evaluated model track-etched membranes and characterized them for pore density and pore size
- Conducted polymer membranes and fabricated them into thin-film composites
- Constructed and tested ion transport cells and performed preliminary measurements to characterize the response of both the track-etched membranes and the polymer membranes.

Experiments are ongoing to provide a systematic series of data on both idealized and real membranes for input into the modeling effort. Our modeling activities focused on detailed models of a single, idealized pore that will serve as the basic building block for membrane and system level models. Pore diameters are on the order of nanometers and therefore require particle-based methods to accurately simulate ionic transport.

We are using molecular and Brownian dynamics to model particle-particle interactions and gain detailed insight into ion partitioning effects at the pore mouth. A few pore diameters away from the nanopore, the relevant physics can be well represented using a continuum formulation, which relies on the Poisson-Nernst-Planck equations and is computationally much faster than the particle-based methods.

We coupled the continuum formulation to the particle simulations using a handshake zone that exchanges information between the two models needed for their respective boundary conditions. We implemented the three aspects of the single-pore model – particle-based formulations, handshake zone, and continuum formulation – and are testing them in simulations of ion transport through nanopores with an applied voltage and concentration difference across the membrane.

Significance

Both the experimental and computational work performed as part of this project is generating a better understanding of the fundamental aspects of ion transport through nanopores. Increased understanding and development of a predictive modeling capability will allow the science-based design and optimization of nanoporous membranes for water desalination and purification with dramatically improved efficiency and selectivity.

Besides the application to water treatment, the primary focus of this project, the result of this work will be highly relevant to the understanding of transport through membranes for fuel cells and through nanopores in general.

Novel Virus Coagulants for Water Treatment and Biomolecular Structural Science

93559

M. D. Nyman, J. M. Bieker, C. E. Ashley

Project Purpose

The purpose of this project was to explore novel coagulant chemistries for removal of pathogens from drinking water and to optimize alumina-based coagulant chemistries. Of more fundamental interest, we are investigating the mechanism of pathogen-flocculent interactions by creating model ordered arrays consisting of pathogens (viruses), inorganic clusters, and amphiphiles such as surfactants.

From these ordered arrays, we hope to obtain an understanding of both the short-range interactions (atom-atom contact at the interface between inorganic clusters and biomolecules) and long-range ordering. Through this project we aim to produce fundamental science that can be applied to producing potable water either at municipal plants or at-the-source applications, or for wastewater treatment.

FY 2006 Accomplishments

Since the interfacial interactions between the inorganic clusters and the viruses likely play an important role in the efficacy of virus sequestration and flocculation, it is important to compare the behavior of viruses with different surface chemistries. We tested several groups of coagulants for efficacy of sequestering four different viruses: enterovirus, influenza, corona virus, and MS-2 bacteriophage. Enterovirus and corona viruses are waterborne; influenza is not waterborne but is representative of enveloped viruses; and the first two are nonenveloped. The MS-2, also a non-enveloped virus, was used for characterization of flocs since it is noninfectious to humans.

As we expected, the highly charged inorganic clusters (with their added surfactants) were more effective for sequestration of nonenveloped than for enveloped viruses, which supports our hypothesis that interaction between the highly charged clusters and the charged regions of the virus coat plays a significant role in virus sequestration. All cluster-surfactant systems we investigated were effective in reducing the virus concentration by 3-9 log. For the Al_{13}^{7+} inorganic cluster (the active ingredient in commonly used alum flocculation water treatments), we investigated using base (NaOH), sodium dodecylsulfate (SDS), and a mixture of SiO_4 plus dodecyltrimethylammonium bromide ($Si\text{-}C_{12}^{+}$) as the added surfactant or flocculant. Surprisingly, the $Si\text{-}C_{12}^{+}$ flocculant was more effective than the anionic SDS for virus sequestration and precipitation.

We also explored the use of anionic polyoxometalates as the inorganic cluster, namely $[Nb_6O_{19}]^{8-}$, $[SiW_{11}O_{39}]^{8-}$, and $[PW_{12}O_{40}]^{3-}$. The surfactants coupled with these clusters included $Si\text{-}C_{12}^{+}$, $Si\text{-}C_{12}^{+}$, and cetyltrimethylammonium chloride/bromide (C_{16}^{+}). We are still collecting data, but thus far the $Si\text{-}C_{12}^{+}$ or C_{12}^{+} alone again appears to be most effective. Dynamic light scattering data showed the Al_{13}^{7+}- $Si\text{-}C_{12}^{+}$ and $[SiW_{11}O_{39}]^{8-}$ C_{12}^{+} systems to form the largest aggregations in solution, and correlating with an effective flocculation.

Using the noninfectious MS-2 virus, we formed cluster-surfactant virus precipitates to investigate the self-assembly of such arrays. Thus far we have learned that the lamellar structure the cluster-surfactant phases have without the virus is destroyed by the presence of MS-2, suggesting that the virus is enveloped into the precipitate and the long-range order is disrupted.

Significance

This work is applicable to Sandia's water initiative and also to biosciences and homeland security. Certain aspects of this project are fundamental in nature and will be published, accomplishing scientific credibility for Sandia. In the first year, the results of this work have already been leveraged into proposals to the Awwa Research Foundation and the Department of Defense, the latter including a potential collaboration with University of Illinois.

We presented our work at the Materials Research Society Spring Meeting (April 2006), where it was nominated as one of the top five talks of interest to the general public.

Refereed Communications

M. Nyman, J.M. Bieker, J. Bixler, and M.S. Kent, "Inorganic Cluster-Amphiphile Coagulants and Surfaces for Pathogen Removal from Water," presented at the Materials Research Society Spring National Meeting, San Francisco, CA, April 2006.

A Demonstration of Advanced Transparency at the Monju Fast Breeder Reactor

93561

C. M. Mendez-Cruz, V. D. Cleary, J. Reynolds, E. D. Vugrin, K. W. Vugrin, D. L. York, S. A. Caskey

Project Purpose

A key objective to the global deployment of nuclear technology is maintaining nuclear transparency among international communities. Proliferation resistance features are critical to building trust and confidence in a global nuclear future. International Atomic Energy Agency (IAEA) safeguards have been effective in minimizing opportunities for diversion; however, recent changes in the global political climate necessitate implementation of additional technology to ensure the immediate detection of proliferation.

For a variety of reasons, nuclear facilities are becoming increasingly automated and will require minimum manual operation. An automated system generates real-time data that can be used to track and measure the status of the process and material at any given point in time.

Sandia has developed a framework designed to monitor plant process data and quantitatively measure proliferation risk continuously. This framework can lead to greater transparency of nuclear fuel-cycle activities and will support multinational alternatives envisioned in the next 50 years. This research is not intended to replace existing IAEA protocols, but rather improve current transparency ideology.

This real-time proliferation assessment framework must be demonstrated. A working agreement exists between Sandia and the IAEA. Through this collaboration, we intend to demonstrate fuel cycle transparency, first at a Fuel Handling Training Model at the International Cooperation and Development Training Center in Japan, and later at the Monju Fast Reactor.

FY 2006 Accomplishments

Sandia and the Japan Atomic Energy Agency (JAEA) entered into a cooperative agreement to demonstrate a methodology capable of assessing proliferation risk in support of overall plant transparency. The Advanced Fuel Cycle Transparency Framework was developed and published in a SAND report. The framework is being implemented at the Fuel Handling Training Model designed for the Monju Fast Reactor at the International Cooperation and Development Training Center of the JAEA. Using the training facilities of Monju, we are instrumenting the training model and developing the technologies necessary to prove the Advanced Transparency Framework that was proposed.

A technological assessment for hardware and software requirements was completed and implementation is in process. Background work for the secure communications (VPN) between Monju and Sandia was completed, including the development of a Sandia VPN policy to reduce vulnerabilities related to external connections. Communications were activated and tested summer 2006, and the Sandia Monju laboratory was set up. The Transparency Toolbox system collects and aggregates the data. The technical designs for communication between the toolboxes (Monju and Sandia sites) are developed, and the corresponding databases are under construction.

The Transparency Analysis Software analyzes the data to calculate real-time proliferation resistance. The functional designs for this software have been developed, and the software is under construction. A demonstration of the software, simulating one operation from the training model, is scheduled to be presented during the Institute of Nuclear Materials Management Annual Meeting 2006. The conceptual framework for assessing proliferation risk, which will be integrated into the software, has been developed and will be refined in cooperation with JAEA partners in October 2006.

Significance

This project demonstrates the usefulness of automated plant process data to continuously assess proliferation risk. The team established a laboratory capable of acquiring data continuously, with workstations in Sandia and Japan. The collaboration between Sandia and the JAEA allows for initial demonstration. Future customers of a continuous, quantitative measure of proliferation risk include the IAEA, the NNSA Office of Nonproliferation and International Security, and the Department of State.

Refereed Communications

T. Love, G. Rochau, D. York, and N. Inoue, "A Framework and Methodology for Nuclear Fuel Cycle Transparency," Sandia Report SAND2006-0270, Albuquerque, NM, 2006.

Other Communications

G. Rochau, C. Mendez, and D. York, "Advanced Remote Monitoring for Reprocessing System," in *Proceedings of the Institute of Nuclear Materials Management 47th INMM Annual Meeting*, July 2006, CD-ROM.

N. Inoue, T. Irie, T. Kitabata, C. Mendez, G. Rochau, and D. York, "A Demonstration of Advanced Nuclear Fuel Cycle Transparency Concepts," in *Proceedings of the Institute of Nuclear Materials Management 47th INMM Annual Meeting*, July 2006, CD-ROM.

G. Rochau, D. York, and C. Mendez, "Nuclear Fuel Cycle Transparency: An Approach to Support the Global Deployment of Nuclear Power," *International Atomic Energy Agency (IAEA) International Safeguards Symposium on Addressing Verification Challenges,* Vienna, Austria, October 2006.

N. Inoue, T. Irie, T. Kitabata, G. Rochau, D. York, and C. Mendez, "A Demonstration of Advanced Nuclear Fuel Cycle Transparency Concepts – International Forum," to be published in *International Atomic Energy Agency (IAEA) International Safeguards Symposium on Addressing Verification Challenges,* Vienna, Austria, October 2006.

C. Mendez, G. Rochau, D. York, and V. Cleary, "Giving Transparency Concepts a Facelift: Bridging the Gap Between Old and New," to be published in *International Atomic Energy Agency (IAEA) International Safeguards Symposium on Addressing Verification Challenges,* Vienna, Austria, October 2006.

Reliability of Passive Safety Systems

93562
D. G. Robinson, C. B. Atcitty, J. V. Zuffranieri

Project Purpose

Advanced nuclear reactor designs must be designed for long core life and high fuel burn-up, which will pose new challenges for monitoring the state of the highly integrated and largely inaccessible components within the primary reactor vessel. As reactors age, characterizing and predicting the internal operational performance (e.g., vibration/flow characteristics) will be critical for determining maintenance strategies and early identification of safety and reliability issues.

This project investigates an approach that provides the capability to combine information from in situ monitoring sensors within a reactor with maintenance and repair histories to characterize the current probability of a failure event. The approach is based in Bayesian probability theory and provides the capability to predict the probability of failure at some future time.

Degradation in reactor performance can result from stresses related to aggressive thermal and mechanical environments, stress corrosion cracking, fatigue, and so on. Decisions regarding the current and future health of the system can be critical in maintaining a safe and efficient operation of the reactor. Prediction of future failure events can provide a basis for effective planning of preventive maintenance.

The proposed approach seeks to apply Bayesian concepts similar to those used in medical monitoring. System-based medical monitoring uses Markov chain Monte Carlo methods to deliver estimates of the state of health of a process based on data taken from the process in real time. By providing and constantly updating a statistical prediction of the probability of a pending failure, this system can provide operators a warning based on the likelihood of surpassing a minimum risk threshold. Moreover, since the method allows for the necessary data to be collected while the reactor is online and merged with offline failure information, the expected loss associated with premature reactor shutdown is minimized.

FY 2006 Accomplishments

Secured Laboratory Data

We constructed a new set of laboratory experiments; are planning new experiments over a variety of temperatures, relative humidity (RH), and corrosive salt concentrations (NaCl); and are collecting data for 80 °C, 40% RH, and 20 g/cm3 NaCl.

Reviewed Existing Methodologies

We reviewed considerable research conducted under the auspices of the Nuclear Regulatory Commission (NRC) and the International Atomic Energy Agency. In an effort to accumulate information on previous studies, we established contacts with researchers at various universities under contract with the NRC (e.g., University of Michigan, University of Tennessee) and are obtaining permission from NRC to access their results as well as the data already collected. While the data from the laboratory experiments will be extremely beneficial in validating the models for real-time assessments, existing data from in situ sensors will provide a level of credibility to our results. Two student interns from nuclear engineering programs will spend the summer investigating and documenting previous research efforts.

Measured & Extended Approach to Include Prediction

We completed simple models involving a single covariate (NaCl concentration) and single response variable (resistance), and efforts are under way to investigate more complex models. Expansion of the original tasks in this area is currently a major focus, and a research contract with the University of Minnesota is in place to help push the envelope even further.

Documentation in Progress
We submitted a paper titled “Simultaneous Materials Degradation and Failure Rates Estimation Using Bayesian Methods,” by D. Robinson and J. Zuffranieri, for the 8th American Institute of Aeronautics and Astronautics Nondeterministic Approaches Conference.

Significance

The methodology has potential application for a number of Sandia customers, e.g., US Army, and has the potential to change how aging/degradation issues are addressed in characterizing the nuclear weapons stockpile. For example, while considerable effort is expended on characterizing the physical phenomenon associated with materials degradation, the resulting models are not considered when reliability models are rolled into a system assessment.

A variation of the approach proposed here could be used to objectively include laboratory and computer modeling results. The result would be an increased confidence in the stockpile reliability, better decisions regarding limited stockpile test resources, and quicker response to aging problems that might arise that pose increasing threats to both stockpile reliability and safety.

Successful completion of this research would result in Sandia’s being at the forefront of a technology critical to the success of next-generation reactor development, and that could have immediate application for critical components within existing and next-generation reactors. The possibility exists that the methods could be quickly implemented in hardware and lead to outside interest in supporting future research in this area.

The research will also support Sandia in maintaining its position as the leader in risk assessment methods and the reliability characterization of complex systems. The approach proposed here is a natural extension of the direction that the risk community is already moving in relative to probabilistic risk assessment.

Water-Splitting Nanodevices for Solar Hydrogen Production

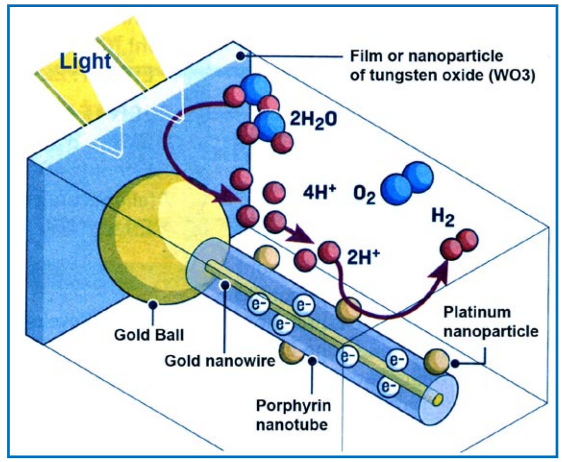
93563

J. A. Shelnutt, C. J. Medforth, R. B. Diver Jr., F. B. van Swol, J. E. Miller, K. R. Zavadil, Z. Wang

Project Purpose

The purpose of the project is to demonstrate the fabrication of multicomponent, self-assembled nanoscale devices for solar hydrogen production and to investigate their functional properties.

These devices use sunlight to produce hydrogen and oxygen by direct splitting of water via two separate photochemical reactions coupled together electrically at the nanoscale. The proposed nanodevices exploit nanoscale architectural control to optimize the sequential processes of light collection, charge separation, and electron transport that are required for efficient water splitting. The construction of these water-splitting nanodevices is made possible by our breakthroughs in the synthesis of robust photoactive porphyrin nanostructures such as nanotubes, nanofibers, and nanospheres by ionic self-assembly of two oppositely charged porphyrin subunits.



A prototype water-splitting nanodevice based on the porphyrin nanotubes, which may function as one of the two coupled photocatalysts required to split water into hydrogen and oxygen. In the nanodevice shown, tungsten oxide photocatalytically evolves oxygen using ultraviolet and blue light, while producing electrons that move through the gold conductor to the nanotube. There, the electrons are promoted by the photoactive porphyrin nanotubes to high enough energy to reduce water to hydrogen using visible light.

We can use the novel photocatalytic and/or light-harvesting properties of the porphyrin nanostructures to generate the metallic components required for catalytic hydrogen evolution and to grow the electrical interconnects between the hydrogen- and oxygen-producing components of the nanodevices. By making and studying these nanodevices, we are gaining the understanding of the photophysical and electronic processes and intercomponent interactions that is necessary to exploit the advantages of the nanoscale (e.g., short distances between excitation and electron-collection points).

Our primary goals are to develop general methods for synthesizing the nanostructures and nanodevices; characterize their structural, electrical, and optical properties; and measure their water-splitting activity. We made substantial progress in the areas of fabricating and testing a porphyrin-nanotube-based hydrogen-evolving component and in the use of porphyrin nanorod bundles as light-harvesting components of the nanodevices. We are also combining experiments and computer simulations to develop a mature understanding of the molecular interactions and the relevant electron- and energy-transport mechanisms. These studies could provide the fundamental breakthroughs necessary for efficient solar water splitting and facilitate the establishment of a hydrogen-based economy.

FY 2006 Accomplishments

We made steady progress in creating and investigating hydrogen-generating solar nanodevices based on two different design approaches for solar water splitting. We fabricated and structurally characterized the hydrogen-evolving part of these water-splitting nanodevices and evaluated three of these types of nanodevices with regard to their relative rates for hydrogen production. The hydrogen-producing component in these nanodevices is based on porphyrin nanostructures that were discovered at Sandia and synthesized by our research team. We found that the porphyrin nanostructures possess diverse energy- and electron-transfer capabilities, which can be exploited in our design strategies. In particular, we identified and demonstrated two general methods for using the porphyrin nanostructures in water-splitting nanodevices.

In the more bioinspired approach, the porphyrin nanostructure serves as the light-harvesting component. When coupled to a reaction-center chromophore, separate catalysts for water oxidation and proton reduction, and suitable electron-relay components, the porphyrin nanostructure efficiently transfers excitation energy to the reaction center where charge separation occurs, ultimately generating hydrogen. The highest hydrogen yields to date have been obtained using this approach in a nanodevice in which platinized porphy-rin nanorod bundles or nanospheres serve as the light- harvesting component and anthracene carboxylic acid is the reaction center molecule. Hydrogen is rapidly evolved from platinum nanoparticles adsorbed onto the porphyrin nanorod bundles or nanospheres. Methylviologen is used as an electron relay between anthracene acid and the platinum particles.

In the second type of water-splitting nanodevice, the platinized porphyrin nanostructure serves as a photocatalyst (essentially a narrow band-gap semi-conductor) that carries out the hydrogen-evolving half of the reaction. In this case, the porphyrin nano-structure has to be capable of electron transfer to the hydrogen-evolution catalyst (platinum nanoparticles). We made progress in constructing this type of device using porphyrin nanotubes containing gold nanowires in the cores of the tubes with platinum nanoparticles on the outer surface. We increased the yield of hollow porphyrin nanotubes for this type of water-splitting nanodevice. We also verified preliminary results that showed hydrogen evolution from porphyrin nanotubes with adsorbed platinum nanoparticles when irradiated with visible light. Demonstrating that both approaches we use are viable for developing hydrogen-producing nanodevices, we began the initial steps required to couple the hydrogen-evolving components of the nanodevice to the oxygen-producing components.

Significance

The production of efficient nanodevices for solar water-splitting is closely related to Sandia's energy, weapons, and nonproliferation missions. New energy sources based on simple self-assembly methods can replace petroleum fuels if solar efficiencies in the 10 percent range for production of hydrogen can be attained. Commercial solar hydrogen cells, which are composed of similar materials, have solar efficiencies of greater than 10 percent for hydrogen production. These cells are based on inexpensive crystalline III-VI semiconductor materials, titania, and organic dyes similar to the materials used in our water-splitting nanodevices.

Our approach to making water-splitting nanodevices can potentially enhance the efficiency of these types of solar hydrogen cells by taking better advantage of the efficiencies provided by the nanoscale size, such as enhanced stability and nanometer distances between charge or exciton creation and collection points. For nuclear weapons and nonproliferation applications, continued reductions in the size of electronic and photonic devices and power sources and their increased reliability are key goals. The potentially new types of devices that could result from our bottoms-up approach used in making the water-splitting nano-devices are expected to demonstrate a path forward in the development of similar nanoelectronics, nano-photonics, and small power sources.

Refereed Communications

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H. Wang, Y. Song, C.J. Medforth, and J.A. Shelnutt, "Interfacial Synthesis of Dendritic Platinum Nano-shells Templated on Benzene Nanodroplets Stabilized in Water by a Photocatalytic Lipoporphyrin," *Journal of American Chemical Society*, vol. 128, pp. 9284-9285, July 2006.

Y. Song, W.A. Steen, D. Peña, Y. Jiang, C.J. Medforth, Q. Huo, J.L. Pincus, Y.Qiu, D.Y. Sasaki, J.E. Miller, and J.A. Shelnutt, "Foamlike Nanostructures Created from Dendritic Platinum Sheets on Liposomes," *Chemical Materials*, vol. 18, pp. 2335-2346, May 2006.

Y. Song, Y. Jiang, H. Wang, D.A. Peña, Y. Qiu, J.E. Miller, and J.A. Shelnutt, "Platinum Nano-dendrites," *Nanotechnology*, vol. 17, pp. 1300-1308, March 2006.

Development of Nanostructured and Surface Modified Semiconductors for Hybrid Organic-Inorganic Solar Cells

93564

J. W. Hsu, D. S. Ruby, T. R. Guilinger, P. G. Clem

Project Purpose

The purpose of this project is to understand critical factors that affect the performance of hybrid organic-inorganic solar cells. With production increasing at a rate of more than 20 percent a year over the last 15 years, solar energy conversion is increasingly being recognized as one of the principal ways to meet future energy needs. Hybrid organic-inorganic solar cells (SCs) are attracting particular interest because of the potential for low-cost manufacturing (printable technology) and for use in new applications, such as power sources for "future force warriors," architectural integration, and disposable field-deployed sensors.

Key materials advantages of these next-generation SCs over conventional semiconductor SCs are in 1) design opportunities (since the different functions of the SCs are carried out by different materials, there are greater materials choices for producing optimized structures) and 2) tandem cell manufacturability (technically easier and cheaper to build to achieve higher efficiencies).

We propose to apply and extend our capabilities in the solution synthesis of patterned, hierarchically nanostructured, semiconducting oxide arrays to this solar energy technology. We are teaming with the National Renewable Energy Laboratory (NREL), an established leader in photovoltaic (PV) and organic PV technologies, to draw upon their expertise in electroactive organic materials (hole conductors) and device fabrication. Having already developed the ability to grow a wide range of nanoengineered structures of ZnO and TiO_2 (semiconductors of choice for PV electron acceptors), we will synthesize and characterize arrays of these materials with compositions, nanostructures, and surfaces optimized for this application.

FY 2006 Accomplishments

We focused on ZnO nanorod arrays as the electron transporter and poly-(3-hexylthiophene) (P3HT) as the light-absorber and hole transporter in hybrid organic-inorganic solar cells. We gained understanding and control over growing oriented ZnO nanorods with sub-50 nm diameters on various substrates. We developed novel approaches to infiltrate commercially available P3HT in the narrow spacing between ZnO nanorods. We investigated the substrate, the solvent used for casting P3HT, and the subsequent annealing conditions in order to optimize ordering in the P3HT, infiltration into dense ZnO nanorod arrays, and electron transfer at the ZnO-P3HT interfaces. We also explored other methods to infiltrate P3HT and to chemically modify ZnO surfaces for more efficient electron transfer from P3HT to ZnO.

An important component of this project is the collaboration with Dr. Dave Ginley's group at NREL. NREL efforts, funded by their Director's Discretionary Research and Development program, focus on testing devices and developing new organics for the hybrid solar cells, which complement Sandia's efforts. We sent four batches of Sandia ZnO nanorod arrays and P3HT infiltrated ZnO nanorod arrays to NREL for device fabrication and testing (capabilities that Sandia does not currently have).

Significance

This project supports DOE's national security mission by developing new technologies for sustainable, future energy generation. This project will lay groundwork for Sandia to be at the forefront of using nanostructures for energy harvesting. This work leverages activities in emergent properties of nanocomposites in the Complex Functional Materials thrust of DOE's new Center for Integrated Nanotechnologies. By applying and extending our capabilities in hierarchically nanostructured semiconductor arrays, we will strategically position Sandia in the growing field of hybrid PVs. The partnership with NREL further enables us to access improved organics and established PV characterization facilities.

Refereed Communications

Y.J. Lee, T.L. Sounart, D.A. Scrymgeour, J.A. Voigt, and J.W.P. Hsu, "Control of ZnO Nanorod Array Orientation Synthesized Via a Two-Step Seeded Solution Growth Process," submitted to *Journal of Crystal Growth*, September 2006.

Other Communications

Y.J. Lee, T.L. Sounart, E.D. Spoerke, D.C. Olson, D.A. Scrymgeour, J.W.P. Hsu, and J.A. Voigt, "Structure Control in ZnO Nanorod Arrays for Photovoltaic Applications," presented at MRS 2006 Spring Meeting, San Francisco, CA, April 2006.

Y.J. Lee, D.C. Olson, E.D. Spoerke, D.A. Scrymgeour, J.A. Voigt, and J.W.P. Hsu, "Orientation Control and Surface Treatment of ZnO Nanorod Arrays for Organic-Inorganic Hybrid Photovoltaics," presented at Electronic Materials Conference, State College, PA, June 2006.

D.C. Olson and J.W.P. Hsu, "Nanocrystal-Based Next-Generation Photovoltaics," presented at IC Postdoctoral Research Fellowship Colloquium, Washington, DC, April 2006.

GNEP Technology Systems Study

104984
V. Tikare, P. Yang, E. A. Holm, T. Y. Chu, R. A. Causey, B. L. Spletzer, C. V. Robino, M. S. Derzon, L. N. Brewer, T. M. Nenoff, J. G. Arguello Jr., M. B. Boslough, W. R. Wampler

Project Purpose

The purpose of this "concept definition" project was to assess the science and technology needs for the proposed Global Nuclear Energy Partnership (GNEP). GNEP seeks to reduce radioactive waste, reduce carbon dioxide emissions, prevent proliferation of nuclear weapons, and provide a safe, reliable, and abundant source of nuclear energy. It will accomplish this by reprocessing light water reactor spent fuel and burning the transuranics in an advanced burner reactor.

The science and technologies necessary for the deployment of GNEP are challenging and exciting. The GNEP systems study brought together a group of staff members with varied technical backgrounds to identify the scientific questions and assess candidate approaches for resolution. This work will enable Sandia to apply its extensive scientific capabilities to solving the technical issues in GNEP.

FY 2006 Accomplishments

A systems analysis of the proposed GNEP system was conducted to assess and evaluate its science and technology issues. A small team of experts, staff members with diverse technical backgrounds, was formed. This team participated in a two-day exercise of structured brainstorming, termed "ideation." On the first day, the team heard presentations from nuclear energy experts who explained the motivation and goals of GNEP and the current state of fast reactor technology, reprocessing technology, and reactor fuel fabrication and performance. The presentation session was followed by a brainstorming session to generate ideas. Some of these ideas were chosen for further investigation to develop into possible research and development projects to support GNEP.

The science and technology needs this team identified were:

- develop and characterize advanced waste forms for Cs, Sr, I, and Tc
- develop new generation fuel systems that incorporate advances in metals and ceramics processing, microstructural tailoring, and characterization
- apply advanced simulation that incorporates high-fidelity models to simulate fuel and reactor performance
- apply sensors, monitors, and remote handlers to run reprocessing and reactor facilities.

Significance

By participating in the study, many key staff members learned of the GNEP program and are in an excellent position to contribute to the technical work that Sandia anticipates as this program grows in future years.

Systems Modeling and Analysis of Transportation Fuels

105673

S. K. Griffiths, G. W. Kuswa, E. B. Stechel, S. H. Conrad

Project Purpose

The purpose of this short, "concept definition" project was to identify a technical path forward for Sandia's development of a broad systems-analysis capability in the area of transportation fuels that is consistent with and builds on Sandia's strategic objectives. Our goals were to identify the key technical questions that need to be addressed; define the systems modeling, simulation, and analysis capabilities required to answer these questions; and lay out the first steps to developing such capabilities. We addressed these goals mainly through a large internal workshop, bringing together a broad cross section of Sandians from the engineering sciences community, systems analysis groups, and current transportation fuels projects.

FY 2006 Accomplishments

Nearly 40 staff and managers from throughout the labs attended the transportation fuels systems analysis workshop in Albuquerque on August 29, 2006. We focused on questions that need answering, the capabilities needed to answer them, and first steps forward. To address these questions, we broke into four groups focused on biofuels, synthetic fuels, fuel efficiency, and conventional petroleum fuels.

In aggregate, these groups generated several hundred relevant questions ranging from cost and production capacity to environmental impact and water use. Risk in developing new technologies, infrastructure needs, competition between technologies, and the course of adopting new production technologies were also common themes across the four focus areas.

For all four focus areas, the capabilities needed to answer the questions fell into five general categories:

- thermodynamic and chemical engineering process models
- financial and risk models
- competitive resource use models
- database management and data verification
- a high-level framework by which these various dynamic subsystems models communicate.

Specifics differed within the four focus areas, but production cost, infrastructure requirements, and net carbon dioxide emissions were prevalent themes in all.

First steps forward proved to be a challenging topic and, in the end, was not satisfactorily resolved. As accurately described by one participant, "this is a complicated mess." The high-level framework was viewed as an obvious starting point as it provides a foundation from which to build more detailed models describing costs, emissions, and resource requirements. However, this approach will take considerable time to develop even a basic capability to answer current questions like, "Does synthetic fuels production make sense and, if so, under what conditions?" and "What are the water requirements for widespread adoption of biofuels?"

The questions are better addressed through focused technology assessments, but starting with such assessments does not directly set us on a path to develop broad capabilities applicable to a range of questions, nor does it necessarily provide us with new tools that are applicable to building this capability. Additional work is thus needed in how best to begin.

Significance

The intent of this project was to identify a technical path forward for Sandia's development of a broad systems analysis capability in the area of transportation fuels. The significance to the broader science and technology community will unfold only as we build and use the capability. The immediate target audience is internal to Sandia. We attempted to frame a very soft, ill-defined technical undertaking in a manner that makes possible the initiation of productive follow-on projects that collectively help build a broad capability consistent with the Laboratories' strategy. In this endeavor, we made significant progress in bringing together the technical expertise and discussing the challenges but did not fully succeed in laying out a clear path forward.

Engineering Sciences

Ray Model of High-Frequency Cavity Scarring

67064

L. K. Warne, R. E. Jorgenson, J. D. Kotulski, M. B. Higgins

Project Purpose

The development of high-frequency electromagnetic sources leads to a requirement to understand the performance of systems subjected to short-wavelength coherent radiation. Interaction with these systems is usually a two-step process: radiation enters the system interior (cavity) then couples to interior antennas or circuits. These frequencies are usually beyond what numerical simulations can handle directly.

In recent years we analyzed these problems using concepts developed in quantum mechanics (the approach to the classical limit). Given that the cavity is irregularly shaped, the eigenfunctions (modes) are assumed to be normally distributed. Microwave experiments showed that deviations from this simple assumption exist when the cavity boundary is of sufficient regularity to support periodic ray trajectories. This is called scarring. Weapon cavities are typically more complex than the simple shapes studied in these experiments, but we do not know to what extent these deviations persist as the boundary becomes more complex.

In this project we proposed to use a modification of high-frequency ray techniques to examine the connection between boundary topology and scarring (localized modal field enhancements). Successful completion of this project will provide a means of modeling high-frequency electromagnetic problems that are beyond the range of current simulations. The theory developed will provide an understanding of modal field enhancements from boundary geometry that will allow better facility designs (mode-stirred chambers). It also will allow better interpretation of probe measurements and predictions of electromagnetic compatibility, covering the entire range of electromagnetic requirements. Finally it will provide underpinning, and possible modifications, for electromagnetic field level controls in explosive handling facilities (V curve).

FY 2006 Accomplishments

- Odd problem in two dimensions (2D)
- Asymmetric problem in 2D
- New projection operator and eigenfunction representation
- Integral of square of field along scar in stadium cavity
- Higher-order terms (focal shift)
- Unstable scar statistics were formulated in three-dimensional (3D) axisymmetric geometry
- Experimental cavities and measurement apparatus were constructed, and 3D measurements made
- 3D ray tracing setup and exponents were calculated.

During FY 2005 we had some success in treating time-harmonic, 2D cavity scars (unstable localized field enhancements along a trajectory between reflectors), including the case with concave surfaces. However, at the beginning of the present year, several remaining issues needed to be addressed.

The first issue had to do with the limitation of treating only the even symmetry with respect to the scar. First, we treated the odd symmetry case in the bow tie geometry. We then showed that the general asymmetric cavity geometry is described by a combination of the even and odd scar results interlaced in frequency. Simulations with the boundary element code in EMPHASIS were carried out for these asymmetric cavities and compared to the theory.

The next issue had to do with the definition of the projections taken along the scarred orbit, which define the amplitude of enhanced field components along the scar. We replaced the original Fourier components used in the convex bow tie cavity with more physical scar projections in the concave stadium cavity. Again, we showed that the behavior of these new scar amplitude definitions were in agreement with the simulations.

With this redefinition of the scar amplitude and the addition of a random plane-wave part to the cavity eigenfunction, we calculated the integral of the square of the field along the scar in the stadium cavity and compared it to simulations.

The final issue was the underpinning for the high-frequency wave solutions. We set up a perturbation series, with the leading term being the high-frequency approximation we examined last year. Higher-order terms can be examined that may give further insight into effects such as the subwavelength focal point shift in the stadium cavity.

We also examined the 3D axisymmetric scar problem. In the high-frequency limit, a scalar formulation of the wave equation can be justified. The scarred eigen-functions in this case are described by a form of the hypergeometric function (rather than the parabolic cylinder functions in two dimensions).

We constructed two sets of cavities to examine fields along scarred orbits in 3D axisymmetric geometry. One involved convex end walls, and the other involved concave end walls. We obtained perturbing conducting and dielectric probes of various shapes to infer field levels from resonant frequency shifts. We carried out several measurements along the scarred orbit.

Finally, we generalized the 2D ray tracing of the previous year to 3D and obtained stability exponents.

Significance

The work will benefit the national security mission by providing a basic understanding of, and means to calculate, how high-frequency electromagnetic waves interact with shielded systems and how antennas and cables respond in such systems. It will improve understanding and design of test facilities such as mode-stirred chambers and associated instrumentation. It will also improve our basic understanding of measurements in such systems.

The demonstrated capability to predict very high-frequency, time-harmonic field enhancements along unstable scarred orbits is an important addition to statistical treatments of chaotic field behavior in cavity environments. This is particularly true for capturing "worst case" penetrations for high-consequence systems.

This demonstrated capability in canonical geometries should be possible to apply to more general systems by integrating it into a general-purpose ray tracing code.

Other Communications

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Noncontact Surface Thermometry for Microsystems

67067
S. P. Kearney, L. M. Phinney, J. R. Serrano

Project Purpose

Microelectromechanical systems (MEMS) components are playing increasingly critical roles in Sandia-engineered systems and within the larger engineering community. MEMS components that have recently undergone evaluation for Sandia applications include: optical switching technologies, vertical-cavity surface-emitting lasers (VCSELs), thermal actuators, and other microscale technologies such as the microChemLab™. The small thermal mass and high relative surface area of MEMS structures renders the thermal response of these systems critical. Meanwhile, the ever-shrinking length scales of these devices revealed different, and often poorly understood, thermophysical behavior from the macroscale.

Characterization of the thermophysics of MEMS structures requires, first and foremost, an accurate knowledge of the device temperature field and its gradients. Historically, Sandia has not possessed the capability for nonperturbing temperature diagnostics with the micron-scale spatial resolution required for experiments on working MEMS devices. This project is meeting Sandia's need for microscale thermal measurements through the development and application of laser-based diagnostics for MEMS thermometry.

We selected the temperature diagnostics based on a list of objectives for thermal MEMS diagnostics, which included the following:

- Spatial resolution on the order of 1 micron or better
- Ability to probe "as-fabricated" MEMS without changes to device design
- Little or no impact upon MEMS device function
- Multipoint scanning measurements for one-dimensional or even two-dimensional profiling.

Selection of optical diagnostic tools ensures "as-fabricated" testing of MEMS components. Optical methods merely require optical access to the device, so that intrinsic sensors or special dopants need not be added in the fabrication process. We selected Raman spectroscopy and thermoreflectance techniques from a list of candidate optical methods, as these diagnostics meet all four of the objectives listed. Both methods use a low-power, nonperturbing probe laser beam, which does not impact MEMS device function.

The laser wavelength is arbitrary, and we select visible laser frequencies, which permit probe-beam focus spots of 1 micron or better and meet our needs for extreme spatial resolution. Detailed spatial profiles are obtained by simply scanning the probe-laser spot over the device surface, and these methods have been used to obtain detailed maps of temperature profiles on MEMS structures, giving Sandia a much-needed experimental capability for development and validation of thermal MEMS design tools.

FY 2006 Accomplishments

- Used Raman spectroscopy for the first-ever simultaneous measurements of temperature and mechanical stress on a working MEMS structure
- Conducted Raman temperature mapping of the ultrafine source-drain features of a GaN/SiC-fabricated high electron mobility transistor (HEMT)
- Acquired the first-ever spatially resolved validation-quality temperature data for the widely used flexure-style thermal actuator design
- Applied the thermoreflectance technique to MEMS thermal actuators with a quantitative comparison to a Raman standard
- Completed conceptual design and construction of a filtered Raman scattering diagnostic for temporally resolved MEMS thermometry
- Received a "best paper in symposium" award from the Materials Research Society for a presentation titled "Micro-Raman Evaluation of Polycrystalline Silicon MEMS Devices."

Significance

Thermal design issues are paramount in almost every MEMS design. Historically, Sandia has not possessed the experimental capabilities for the high-precision, spatially resolved thermometry that is needed for development and validation of thermal MEMS design tools. This project provided Sandia with experimental tools to support mission-critical microsystems development programs, which will have high future impact on Sandia's national security mission.

This project also contributed several advances to the general research and development community. We provided some of the first-ever validation-quality experimental data needed for the development of active-MEMS actuators, which are currently being pursued by a wide variety of academic, industrial, and government institutions. The experimental tools developed here led to collaborations both inside and external to Sandia, and we expect this to continue after the conclusion of this project.

Refereed Communications

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Other Communications

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High-Speed Interferometric Deformation Measurements

67068
P. L. Reu, B. D. Hansche, G. C. Stoker, J. E. Massad

Project Purpose

The purpose of this project was to create a proof-of-concept widefield laser Doppler velocimetry (WLDV) system. This is a significant and important extension of the more common scanning laser Doppler velocimeter inthat all of the measurement points are taken simultaneously. This fills an important niche in velocimetry where the sample either undergoes transient, non-repeatable motions, or is destroyed during actuation.

Current scanning systems would only be able to measure a single data point during the event. The widefield system, however, can measure thousands of points simultaneously, by imaging the device illuminated by a laser and heterodyning the resulting Doppler signal down into the bandwidth of the camera. We also conducted fundamental investigations into laser speckle and related phenomena.

FY 2006 Accomplishments

We demonstrated an operational WLDV by making measurements of both temporally and spatially varying velocity fields. We assembled a number of hardware and software systems to create the WLDV, including:

- Mach-Zehnder Doppler interferometer optics
- Tracking single-point LDV/modulator combination for automated modulation frequency control
- Quick-view analysis algorithms to immediately check velocity results
- In-depth data analysis algorithms and techniques that convert the Doppler intensity information into a time-varying velocity
- Software to unwrap aliased data to extend the bandwidth of the velocimeter.

We conducted proof-of-concept experiments:

- Time-varying velocity measurement of a pendulum
- Spatially-varying velocity field of a rotating plate
- Time and spatially-varying velocity of an accelerating rotating plate
- Mode shape measurement of a vibrating thin plate.

We used these four experiments for hardware and software development. They show the feasibility of using the imaging velocimeter for experimental measurements.

To aid in the understanding of the physics, we undertook three important related tasks:

- Optimum speckle size study for use with dynamic interferometry, including Doppler and speckle interferometry
- Alias unwrapping as a means of extending the bandwidth of measurement devices
- Development of a speckle simulation program to study speckle fields and their affect on velocity imaging.

In addition, we submitted a technical advance for an optical antialiasing filter.

Significance

The development of the WLDV fills an important niche in velocity and vibration measurement. Current measurement systems scan a single point over a structure to build up the velocity image; the WLDV acquires all of the data simultaneously. This has two immediate measurement advantages that the single point system does not have: 1) the ability to measure a nonrepetitive transient event and 2) usefulness in tests where the device is destroyed upon actuation.

WLDV data density is also typically much higher, on the order of tens of thousands of points as opposed to hundreds for a scanning system. It is also likely that the acquisition and analysis time for an equivalent number of points may be shorter than for an equivalent scanning system.

One application not investigated under this project was using the system to measure displacement rather than velocity. Future work could look at this important application, which could provide a more robust electron speckle pattern interferometer (ESPI) system. Typically, ESPI has been extremely sensitive to parasitic room vibrations that often lead to loss of information. The dynamic measurement nature of the WLDV can potentially alleviate this problem.

Refereed Communications

P.L. Reu and B.D. Hansche, "Widefield Laser Doppler Vibrometer Using High-Speed Cameras," in *Proceedings of the SEM Annual Conference*, June 2006, CD-ROM.

Fundamentals of Nanofluidics

67069
R. H. Nilson, S. K. Griffiths

Project Purpose

Nanoscale transport plays an important role in a broad range of engineering applications, including thin film flows, membrane filtration, colloidal particle motion, microfabrication, and electrokinetic transport in chip-based devices for detection of chemical and biological species. Despite this broad importance, there currently exists no reliable means for simulating flow in domains of micron to nanometer scale.

Traditional continuum models fail to account for the molecular-scale physics of structured fluid layers adjacent to solid surfaces. These layers are of critical importance not only in nanoscale channels of recent interest, but also play a critical role in controlling interfacial processes, electric double-layer phenomena, and the fluid/solid slip velocity in channels of millimeter scale and larger.

Conversely, molecular dynamics simulations, which properly account for these atomistic noncontinuum physics, typically require relatively lengthy computing times even on nanoscale domains and become entirely impractical for the submicron- to micron-scale domains of interest in many applications.

The purpose of this project was to develop a rigorous yet tractable methodology for incorporation of non-continuum atomistic physics into classical continuum modeling of fluid flow and related transport processes in channels ranging in size from a few angstroms upward to the millimeter and larger scales of engineering devices.

We succeeded in developing these multiscale models of flow and transport by combining density functional theory (DFT) with classical continuum mechanics. DFT is used to compute structured near-surface profiles of fluid and ion densities and electrochemical potentials based on Lennard-Jones attractions, hard sphere repulsions, Coulomb forces, and short-range electrical interactions. These DFT profiles provide the basis for calculation of fluid viscosity distributions, surface slip velocities, and electrokinetic driving forces that are then used in solving the continuum transport equations.

An important benefit of this approach is that the non-continuum information derived from DFT and related constitutive modeling can be readily incorporated into any of the sophisticated finite-element computer codes developed over past decades at considerable expense.

FY 2006 Accomplishments

In FY 2004 we developed our hybrid atomistic/continuum modeling approach. In FY 2005, we verified this modeling by comparing our DFT-based predictions of fluid velocities with published molecular dynamics simulations of shear-driven Couette flows and electroosmotic flows in channels ranging from 1 to 20 nm. Also in FY 2005, we developed a two-dimensional DFT model by extension of the one-dimensional model developed in FY 2004. And, we performed and published a comprehensive analysis of electroosmotic pumping devices and demonstrated that their performance is optimal when the lateral channel dimensions are comparable to the electric double layer thickness, typically ranging from 1 to 100 nm.

In FY 2006, we built upon our previous work to accomplished three principal goals:

Influence of Atomistic Physics on Electro-Osmotic Flow
An Analysis Based on Density Functional Theory (accepted by *The Journal of Chemical Physics*).
We used molecular dynamics profiles and charge distributions determined by DFT in conjunction with the continuum Navier Stokes equations to compute electroosmotic flows in nanoscale channels. For a prescribed surface charge, this DFT model predicts larger counterion concentrations near charged channel walls, relative to traditional Poisson-

Boltzmann (PB) continuum modeling, and hence smaller concentrations in the channel center. This shifting of counterions toward the walls reduces the effective thickness of the Debye layer and reduces electroosmotic velocities as compared to classical PB modeling.

Zeta potentials and fluid speeds computed by the DFT model are as much as two or three times smaller than corresponding PB results. This disparity generally increases with increasing electrolyte concentration, increasing surface charge density, and decreasing channel width. The DFT results are found to be comparable to those obtained by molecular dynamics simulation, but require orders of magnitude less computing time, thereby enabling this first comprehensive study of electrokinetic flow in nanochannels.

Charged Species Transport, Separation, and Dispersion in Nanoscale Channels: Autogenous Electric Field-Flow Fractionation
We used numerical modeling to investigate the transport of charged species in pressure-driven flow and electroosmotic flow along nanoscale channels having an electric double-layer thickness comparable to the channel size. In such channels, the electric field inherent to the double-layer produces transverse species distributions that depend on species charge. Flow along the channel thus yields mean axial species speeds that also depend on the species charge, enabling species separation and identification.

For pressure-driven flows, we demonstrated that mean species speeds along the channel are uniquely associated with a single species charge, allowing species separation based on charge alone. In contrast, electro-osmotic flows generally yield identical speeds for several values of the charge, and these speeds generally depend on both the species charge and electrophoretic mobility. This study is the first to provide a quantitative basis for designing a new class of nanofluidic separation devices.

Wall Functions for Incorporation of Atomistic Physics into Continuum Modeling of Electrokinetic Flow
We used wall functions derived from DFT to incorporate noncontinuum atomistic physics into continuum modeling of electroosmotic flow in slit-like channels ranging in width from a few molecular diameters to scales several orders greater.

These wall functions represent deviations of the electrochemical potentials of charged and uncharged species from their nominal continuum values due to Lennard-Jones interactions among fluid and solid molecules, hard sphere repulsions, and noncoulombic electrical interactions. Because these deviations decrease strongly with distance from the charged surfaces, the wall functions computed by DFT for a channel width of 10 or more molecular diameters can be applied to all wider channels using any existing continuum modeling platform.

Significance

This project has provided increased fundamental understanding of nanofluidic phenomena and a unique capability to incorporate noncontinuum atomistic physics into continuum modeling of nano- and macroscale systems. The use of DFT in modeling of noncontinuum physics has facilitated the derivation and very rapid computation of wall functions for incorporation of atomistic physics into the traditional continuum modeling tools developed over many years at the national laboratories and elsewhere. Areas of current and future importance to Sandia's core missions are outlined below.

Homeland Security
The new modeling capabilities developed under this project are needed to design and optimize microChemLab™ and electrokinetic pumping devices that play a central role in Sandia's Homeland Security and Defense (HSD) programs. This rapidly growing HSD effort promises to become a core business area that may also leverage Sandia's nanofabrication technologies developed under the extreme ultraviolet lithography program.

Our work is applicable not only to electro-osmotic and electrophoretic separation devices, but also to transport of colloidal particles, filtration through nanoporous membranes used for water purification, as well as ion transport through biological cell walls.

To gain improved understanding and to optimize performance of these nanofluidic devices, we performed and published under this project comprehensive computational studies of three systems of fundamental importance: electro-osmotic flow in nanochannels, electrokinetic pumping devices, and autogenous separation of charged species in nanoscale channels.

Defense Programs
These new modeling capabilities are also applicable to lubrication of microfabricated devices of current interest for weapon arming and safing. The ability to model these frictional forces becomes increasing important as the device scale shrinks. The performance of heat-pipes and heat spreaders used in cooling microelectronics is also dependent on flow in nanoscale evaporation films at the fluid/solid contact.

Similarly, nanofabrication and replication processes such as injection molding involve the advance of surface wetting fronts and the still unresolved issues surrounding the role of surface slip in controlling dynamic contact angles and propagation speeds. Another potential application of this project involves the storage of gases and electrical energy in nanoporous materials.

Energy
Nanostructured materials such as carbon nanotubes, zeolytes, and aerogels appear to offer great promise for storaging gases at high densities but moderate pressures. Similarly, carbon-based nanoporous electrodes provide the basis for supercapacitors capable of energy densities three to four orders of magnitude greater than conventional capacitors. These advanced materials achieve high storage capacities through extremely high surface areas associated with nanoscale pores. However, these nanoscale benefits come only at the expense of longer charge and discharge times owing to reduced transport rates in the smaller pores.

To balance these opposing requirements requires engineered materials consisting of nanoscale pores that provide capacity and selectivity, connected by a network of somewhat larger microscale channels needed to achieve rapid charge and discharge rates. With these applications in mind, we are building upon the modeling capabilities developed under this project to perform multiscale analysis of multiphase transport (gas, adsorbate, liquid) in nanoporous materials.

Refereed Communications

R.H. Nilson and S.K. Griffiths, "Influence of Atomistic Physics on Electroosmotic Flow: An Analysis Based on Density Functional Theory," to be published in *The Journal of Chemical Physics*.

S.K. Griffiths and R.H. Nilson, "Charged Species Transport, Separation, and Dispersion in Nanoscale Channels: Autogenous Electric Field-Flow Fractionation, to be published in *Analytical Chemistry*.

R.H. Nilson and S.K. Griffiths, "Wall Functions for Incorporation of Atomistic Physics into Continuum Modeling of Electroosmotic Flow," in *Proceedings of the NSTI Nanotech 2006*, pp. 503-506, May 2006.

R.H. Nilson and S.K. Griffiths, "Hybrid Atomistic/Continuum Modeling of Electroosmotic Flow in Nanoscale Channels," in *Proceedings of the Micro Total Analytical Systems 2005*, pp. 235-237, October 2005.

R.H. Nilson and S.K. Griffiths, "Modeling Microscale Flow by Combining Density Functional Theory with Classical Continuum Mechanics," presented at the 8th US National Congress on Computational Mechanics, Austin, TX, July 2005.

Simulating Self-Assembly and Growth of Biological Nanostructured Materials

67070

T. D. Nguyen, D. R. Noble, P. J. in't Veld, D. J. Holdych, M. J. Stevens

Project Purpose

Self-assembly of biomolecules is a promising avenue for developing new materials. Engineering principles for micro/nanofabrication can be discovered from the study of self-assembly phenomena in biological systems. The purpose of this project is to develop a multiscale and multiphysics framework for modeling the processes of self-assembly and growth in a representative biological material.

The mathematical description of growth constitutes a highly complex system that requires mixture theory with coupled transport, reaction, and mechanical deformation processes. The macroscopic phenomena of growth and self-assembly at any given location are driven by microscale processes of transport of reactants and byproducts, and by the nanoscale process of deformation of the tissue's nanostructure. Information from these lower-scale processes must be incorporated into continuum scale descriptions of growth.

FY 2006 Accomplishments

In this final year of the project, we completed the finite element implementation of the continuum mixture theory. The theory incorporated information from micromechanical simulations of stress-driven fluid flow and solute diffusion through constitutive parameters governing fluid and solute transport. We obtained these parameters through direct numerical simulations of an idealized microstructure of a representative collagen tissue, the tendon fiber.

We implemented the balance laws of the mixture theory (linear momentum balance for the mixture and mass balance for individual species) in a staggered implicit integration scheme that enforced convergence for the system of equations. We then applied the mixture theory to simulate stress-driven flow, coupled fluid and solute transport, and coupled transport and mechanical deformation.

We also conducted micromechanical simulations, using the lattice Boltzmann and immersed finite difference methods, of the first two example problems to validate the consistency of the multiscale framework. The results of the continuum finite element simulations and micromechanical simulations showed good agreement. However, the numerical implementation of the mixture theory demonstrated poor convergence and numerical instability for the simulation of transport coupled with mechanical deformation for an incompressible fluid phase.

At the molecular scale, atomistic simulations, using molecular dynamics, were used to simulate the strength of tropocollagen, the triple helix molecule, in tension and tearing. A coarse-grain model of the collagen fibril was also developed and applied to simulations to characterize the stress-strain behavior and stability of the fibril structure.

Significance

We developed a multiscale and multiphysics framework for modeling the coupled processes of transport, chemical reactions, and mechanical deformation governing growth and self-assembly. There are still significant numerical and theoretical issues that remain to be addressed, but we developed a solid foundation for developing a physics-based mixture theory.

The results of this work can have significant impact in the design of nanoscale manufacturing processes that involve self-assembly and the development of bioinspired materials. Extension of this work is being explored for investigating the pretreatment process of recalcitrant cellulose in the production of biofuels.

Refereed Communications

H. Narayanan, E.M. Arruda, K. Grosh, and K. Garikipati, “Biological Growth: Reaction, Transport, and Mechanics,” submitted to *Biomechanics and Modeling for Mechanobiology*.

Other Communications

D.J. Holdych, T.D. Nguyen. D. Noble, and P.A. Klein, “Modeling Nutrient Transport Coupled with Mechanics in Collagen,” presented at US National Congress on Theoretical and Applied Mechanics, Boulder, CO, June 2006.

D.J. Holdych, T.D. Nguyen, D. Noble, and P.A. Klein, “Modeling Nutrient Transport Coupled with Mechanics in Collagen,” presented at World Congress on Computational Mechanics, Los Angeles, CA, July 2006.

Investigation of Liquid Jet Break-Up and Dispersion

79764

R. A. Jepsen, S. C. James, B. Demosthenous, T. J. O'Hern

Project Purpose

Knowledge of dispersed liquid-spray dynamics is necessary for concerns such as fuel ignition leading to over-pressurization. Strategic applications of fuel dispersion include thermobarics and 9/11-style scenarios involving liquid fuel dispersal. Other applications include launch and transportation accident definition and fire suppression system design. In these cases, the creation of a dispersed liquid depends on the break-up of a much larger liquid slug or jet.

Recent modeling and diagnostic efforts as well as phenomena evaluation of liquid dispersion dynamics demonstrated that interactions of liquid jets or slugs in air or on impacting surfaces are poorly understood. This is an important knowledge gap, since the results of the break-up process often determine the initial conditions needed for more robust particle transport models.

While the research is discovery in nature, experiments also focus on obtaining data specific to thermobarics and liquid-slug impacts. This will include development of laboratory-scale techniques to eject liquid slugs such as water or other fuel surrogates using an air gun or pressurized actuator. Specific diagnostics will also be developed and applied to the experiments. These include laser techniques such as particle image velocimetry (PIV), phase Doppler particle analysis (PDPA), high-speed photometrics, and load cells. In the initial stages of the research, we will obtain spatially resolved data in the regions where the liquid continuum transitions to individual droplets.

This research develops novel experimental techniques and provides results that lead to an improved understanding of liquid dispersal phenomena. In addition, theoretical and model development provides the ability to predict dispersal and ignition characteristics of fuels as a function of liquid-slug velocity and impact conditions.

FY 2006 Accomplishments

This project has been extremely successful in the last two years. We conducted a series of experiments at a wide range of Weber numbers (10^2 to 10^8) to gather discovery data and develop supporting models and theory. We applied several diagnostics to obtain both qualitative and quantitative data. Much of this data is well supported by several model simulations. This demonstrates an excellent understanding of the processes investigated.

We successfully implemented and used novel experimental techniques. Results from low Weber number ($We < 10^3$) impact testing match previously published data. In addition, higher-speed testing up to 100 ft/s ($We > 10^6$) revealed important new phenomena in the impact and splashing characteristics.

Significance

The published work from this research describes several new insights on the origins of splashing instability and formation of jets and droplets as well as the effects of air and droplet impact shape on dispersion processes. For example, a discovery from this research not previously known to the droplet impact and spray community is that initial ejection and spreading from the impact point is greatly affected by interactions with the air.

In some cases, the initial ejection speed can be five times greater than the impact speed as it is entrained by the escaping air between the droplet and the impact surface. This is very important in determining spreading distance and spray particle size. This new research is the foundation that could revolutionize the current theory on dispersion processes for applications such as ink jet printing and fuel injection combustion.

A paper we presented, "Diagnostics for High-Speed, Large Liquid-Slug Impact and Dispersion," was nominated for best paper at the Institute for Liquid and Spray Systems 2006 conference.

Insights from this work are being used in fuel dispersion and ignition scenarios for launch safety analysis of radioisotope thermoelectric generator space units.

Refereed Communications

R.A. Jepsen, S.S. Yoon, and B. Demosthenous, "Effects of Air on Splashing during a Large Droplet Impact: Experimental and Numerical Investigations," to be published in *Atomization and Spray*.

R.A. Jepsen, S.S. Yoon, and S.C. James, "Investigation of Break-Up, Splash, and Fingerlike Instabilities for a Large Water-Slug Impact," to be published in *ASME International-IMECE*.

S.S. Yoon, R.A. Jepsen, M.R. Nissen, and T.J. O'Hern, "Experimental Investigation on Splashing and Fingerlike Instability of Large Water Droplets," to be published in *Journal of Fluids and Structures*.

Other Communications

H. Park, S.S. Yoon, R.A. Jepsen, and S.D. Heister, "Modeling Droplet Bouncing and Gas-Pressure Effect on Pre-Impact Condition Using Boundary Element Method," in *Proceedings of the ILASS Americas, 19th Annual Conference on Liquid Atomization and Spray Systems*, p. 8, May 2006.

S.S. Yoon, R.A. Jepsen, M.R. Nissen, and T.J. O'Hern, "Experimental Investigation on Splashing and Fingerlike Instability of Large Water Droplets," in *Proceedings of the ILASS Americas, 18th Annual Conference on Liquid Atomization and Spray Systems*, p. 9, May 2005.

B. Demosthenous, R.A. Jepsen, T.J. O'Hern, E. Bystrom, M. Nissen, E. Romero, and S.S. Yoon, "Diagnostics for High-Speed, Large Liquid-Slug Impact and Dispersion," in *Proceedings of the ILASS Americas, 19th Annual Conference on Liquid Atomization and Spray Systems*, p. 9, May 2006.

R.A. Jepsen, S.S. Yoon, and B. Demosthenous, "Effects of Air on Splashing during a Droplet Impact," in *Proceedings of the ILASS Americas, 19th Annual Conference on Liquid Atomization and Spray Systems*, p. 9, May 2006.

Accelerating DSMC Data Extraction

79765

E. S. Piekos, M. A. Gallis

Project Purpose

In the four decades since Graeme Bird originated direct simulation Monte Carlo (DSMC), it has evolved into a remarkably flexible and accurate means for treating a wide range of molecular and thermal transport problems, from reentry vehicle aerodynamics to phonon transport in silicon films. In fact, for many strongly nonequilibrium problems, DSMC is the only available tool, because it is not subject to the local equilibrium assumptions underlying traditional fluid and thermal simulation techniques.

As is generally the case in engineering, this advantage comes at a cost. DSMC's flexibility stems from its modeling of the Boltzmann transport equation rather than from the more common equations derived from it, such as the Navier-Stokes equations and Fourier's law. The Boltzmann equation, however, has not proven tractable for general problems using continuous variable techniques such as finite elements. DSMC therefore relies on a stochastic scheme for representing the distribution function that is the Boltzmann equation's dependent variable.

Due to this stochastic representation, moments of the distribution, which provide the quantities of interest in most problems, such as number density and temperature, must be recovered via statistical sampling techniques. From a numerical standpoint, this means that additional computational work must be performed to recover the desired quantities from a field that has reached the correct state; a point generally considered as "converged" in other simulation techniques. Indeed, it is not unusual for DSMC simulations to consume the majority of their computation time after the flow field reaches a steady-state.

The difficulty associated with distinguishing a quantity from background noise generally increases if its magnitude is small compared to that of the background. The emergence of microelectromechanical systems (MEMS) has made this a common occurrence in DSMC. In these systems, mean flow speeds are often more than two orders of magnitude smaller than the thermal speeds of the molecules themselves. Furthermore, they often operate at atmospheric pressure, where collisions between molecules are common, also increasing computational expense.

Traditionally, the problem of stochastic noise is overcome with either very long runs or with the use of an excessively large number of particle simulators. This can be an arduous process, because the error is inversely proportional to the square root of the number of samples; so we must, for example, quadruple the number of samples to cut the error in half. This problem is what has given DSMC the reputation of a computationally intensive method.

This problem is not unique to DSMC but is a common problem of all Monte Carlo algorithms. As computers have become more powerful, the importance of Monte Carlo algorithms has increased as researchers increase the fidelity of their simulations. Applications of these techniques can be found in modeling stochastic processes in physics, biology, and even finance. It is therefore not surprising that noise or variance reduction techniques have been extensively investigated and developed. This project presents a critical review of various available techniques applied specifically to DSMC simulations of low-speed flow.

FY 2006 Accomplishments

In the second year of this project, we moved toward more sophisticated methods of noise removal, implementing each method in MATLAB and applying each to the standard test suite used for the filters investigated the previous year. We investigated several spatial methods that made multiple passes through the data: IRON, FCT filtering, and anisotropic diffusion.

Fippel and Nusslin developed the "iterative reduction of noise" (IRON) technique to smooth Monte Carlo-calculated dose distributions for radiation therapy. In the IRON technique, the data points are shifted by an optimization algorithm. Unlike the filters we investigated in FY 2005, which we described in terms of frequency content, the optimization routine in the IRON technique is based on the idea of minimizing local second derivatives as a means for identifying and removing noise. In this work, we also investigated the difference between derivative estimates with varying stencils as a means for identifying noise.

Kaplan and Oran suggested the only technique we examined that was developed specifically for DSMC simulations of low-speed flows. These authors propose using the flux-corrected transport algorithm as a filter to eliminate noise from the simulations. This algorithm uses diffusion, followed by antidiffusion steps intended to protect flow features, to remove noise in a conservative manner.

Anisotropic diffusion, the final spatial method we investigated, was proposed by McCool as a means for reducing noise in synthetic images (scene renderings) in which Monte Carlo algorithms were employed for determining lighting effects. In Kaplan and Oran's method, the diffusive fluxes were controlled by anti-diffusive fluxes of varying strength. In anisotropic diffusion, the diffusion is controlled by allowing the diffusion coefficient to vary in space. This formulation allows great flexibility in how the diffusion is controlled and is much easier to tailor than Kaplan and Oran's scheme.

We investigated the control variates method as a means for removing noise without looking toward neighboring cells. This method uses correlations between variables to eliminate noise. This is attractive because it avoids the danger of smearing physical features so it can be applied more confidently by a novice without affecting the fidelity of the solution. The control variates method is also attractive because it has a firm mathematical basis. Unfortunately, we found that this method is quite weak compared to the spatial methods. We were not able to produce the large error reductions we had come to expect from previously investigated methods. Furthermore, it was difficult to apply this method to variables, such as flow speed, that are not strongly correlated to other quantities. This method did, however, provide a nice framework for understanding how the spatial methods work and why they work so well: the correlation between a variable and its location is far stronger than a variable and another variable – particularly for DSMC simulations where the cells are less than a mean free path in extent.

Significance

Noncontinuum transport effects are significant in an increasing number of technologies of interest to Sandia researchers, as well as to DOE activities across the board. Examples include gas transport in detectors for biological agents, thermal transport in microelectronics, and both gas and thermal transport in MEMS.

DSMC has proven to be a valuable tool in these technology areas, as well as its more traditional role in high-speed gas dynamics, such as the Columbia accident investigation and the Mars Reconnaissance Orbiter insertion analysis.

The standard application of this algorithm can be extremely computationally demanding, however, particularly for low-speed flows such as those found in microsystems. In fact, DSMC activities account for a significant portion of the overall processor cycles consumed at Sandia on a yearly basis.

By allowing shorter sampling times, the research performed in this project could greatly extend the capabilities of tools for modeling noncontinuum effects. By increasing the speed of these simulations, researchers can be more nimble and tackle problems with an expanded scope. The tools we investigated could therefore enable more advanced research and increasingly optimized microsystem designs.

Modal Analysis of Almost-Linear Structures

79766

D. J. Segalman, B. W. Dodson, D. Griffith, J. P. Lauffer

Project Purpose

The purpose of this project was to refine and develop ideas for reducing the size of the mathematical models necessary to capture the dynamics of large structures with modest nonlinearities. Such model reduction would be necessary to facilitate the large number of simulations of such structures needed to predict the range of responses that are associated with such nonlinear systems.

FY 2006 Accomplishments

The most direct approach for solving such problems would be the application of a Galerkin solution using as basis functions the eigenmodes of a reference linear system. We identified the limitation of that approach for problems of localized nonlinearities, as is the usual case in jointed structures, to be that the nonlinear response of those structures involves configurations that do not live in the space spanned by the eigenmodes of the reference linear system. Having identified the source of the difficulties as the inability of the basis vectors to describe the structure kinematics in the vicinity of the nonlinearities, we developed a novel approach to address that limitation. The original basis was augmented by additional vectors that have appropriate discontinuities in the vicinities of the nonlinearities.

The resulting low-order Galerkin models capture the nonlinear response of the full systems with great fidelity. Features to be noted include:

- In the case of small amplitude dynamics, the dynamics are nonlinear, but the gross kinematics are well represented as linear combinations of the eigenmodes of the reference linear system. The augmenting modes are important to get the dynamics right, but their amplitude is small. This is consistent with the behavior seen experimentally.
- In the case of very large amplitude dynamics, energy spills over to high frequencies, but the augmented system is capable of modeling these very nonlinear (macroslip) dynamics as long as there are enough basis functions employed to capture the frequency content.

Significance

This project should facilitate the transformation of nonlinear structural dynamics problems from ones so large that they spend weeks on queue to ones small enough that they can be run within a day or two. This quantitative reduction in problem size should lead to a qualitative change in the way that work is done. Instead of doing full system analysis just once (in the sense of an admiral's test) many calculations employing different estimates for load history or different estimates for joint properties can be run to obtain realistic estimates of actual structural response.

Other Communications

D.J. Segalman, "*Model Reduction of Systems with Localized Nonlinearities*," Sandia report, SAND2006-1789, Albuquerque, NM, March 2006.

D.J. Segalman, D.T. Griffith, and W. Holzmann, "Model Reduction to Stabilize Dynamic Analysis of Jointed Structures Undergoing Macroslip," presented at World Conference on Computational Mechanics, Los Angeles, CA, July 2006.

D.T. Griffith and D.J. Segalman, "Finite Element Calculations Illustrating a Method of Model Reduction for the Dynamics of Structures with Localized Nonlinearities," Sandia report, SAND2006-5843, Albuquerque, NM, September 2006.

Nano/Microengineered Interfaces for Improved Performance and Reliability

79767

E. D. Reedy Jr., J. A. Zimmerman, N. R. Moody, T. D. Nguyen

Project Purpose

Material interfaces play a critical role in determining a layered structure's performance and reliability. Our goal is to develop an understanding of how patterns of small-scale interfacial heterogeneities affect interfacial crack propagation, with an emphasis on understanding how to increase interfacial toughness.

We will perform detailed atomistic and finite element analyses to develop a fundamental understanding of the effect of interfacial topography on crack growth at the nano/microscale. Other atomistic simulations will help guide the development of suitable cohesive zone models for use in the continuum finite element simulations. The finite element simulations will investigate how crack growth along a patterned, non-planar interface can increase the apparent interfacial toughness.

The experimental component of this project will study well-defined patterns of nano/microscale heterogeneities. Lithographic and other techniques will be used to create patterns with various dimensions, shapes, and spacing. Nanoimprint lithography will be used to create patterns with features smaller than 100 nm. We will deposit submicron-thick metal coatings on patterned silicon substrates to create bimaterials with a wide range of properties. The interfacial toughness of these bimaterials will be measured using four-point bend techniques that have been adapted to nanoscale films as well as with stressed metal overlayers. The experimental results will help guide and validate the modeling effort.

FY 2006 Accomplishments

We developed and applied atomistic and finite element techniques to model interfacial separation and also performed experiments to measure the toughness of patterned interfaces.

The finite element modeling effort was focused on two types of interfacial topographies: a rippled interface and a rectangular-toothed interface. These two-dimensional (2D) plane strain models actually define parallel channels with the indicated cross-section.

In addition to the usual potential-based cohesive zone model, we used a novel junction model (inspired by atomic force microscope friction test data) to simulate interfacial crack growth along a tungsten/silicon interface. Calculated results for the rectangular-toothed interface indicate that it has an apparent toughness that is twice the intrinsic interfacial toughness.

We also performed molecular dynamics simulations of a misoriented silicon bicrystal to investigate interfacial failure processes in a brittle material system with an incoherent interface of regular geometry. Results for different combinations of applied shear and tensile loads show that interfacial energy is higher in tension than in shear, and the interfacial energy for the mixed loading case is highest. The calculated interfacial energies for all cases are relatively small, on the order of 1 J/m^2, which is expected for brittle materials.

Our experimental work was focused on measuring the effects of nonuniform chemistries and 2D heterogeneities on interfacial fracture energy. For example, we created a silicon/tungsten interface with an array of parallel channels of rectangular cross-section using wafers patterned with nanoimprint lithography. This interface had a feature size of 100 nm. Interfacial toughness measured using stressed overlayers showed roughly a factor of two enhancement in the apparent toughness of this patterned interface when compared to a flat interface.

Significance

The fundamental understanding developed during this study will enable the development of nano/microengineered interfaces with optimized toughness for critical bonded, coated, laminated, and encapsulated components for defense programs, including those using microelectromechanical systems and emerging nanoscale technologies.

Refereed Communications

H.S. Park, K. Gall, and J.A. Zimmerman, "Deformation of FCC Nanowires by Twinning and Slip," *Journal of the Mechanics and Physics of Solids*, vol. 54, pp. 1862-1881, 2006.

Other Communications

N.R. Moody, M.J. Cordill, J.M. Jungk, D.F. Bahr, and W.W. Gerberich, "The Role of Adhesion and Fracture on the Performance of Nanostructured Thin Film Devices," in *Proceedings of the 16th European Conference on Fracture*, July 2006, CD-ROM.

E.D. Reedy, N.R. Moody, J.A. Zimmerman, T.D. Nguyen, and H.S. Park, "Dependence of Interfacial Toughness on Interfacial Topography," in *Proceedings of the 29th Annual Meeting of the Adhesion Society*, pp. 81-83, February 2006.

Electromagnetic Modeling of Photonic Band Gap Laminates for Tailored Emission

79768

R. E. Jorgenson, W. A. Johnson, D. W. Peters, L. K. Warne, L. I. Basilio

Project Purpose

The goal of this project is to provide the critical computational analysis and design tools for developing the "science" of how photonic band gap (PBG) structures operate so they can be built and installed on military platforms without resorting to excessive experimental prototyping. Interest in PBG structures has increased in recent years because they have the ability to manipulate light in both the optical and infrared wavelength ranges. Coating a hot body with PBG structures, for example, can modify its infrared radiation and mask it from detection.

Intentionally introducing defects in a PBG structure allows the optical band gap to be modified, which leads to the possibility of new optical devices. A PBG structure inhibits photon propagation over a narrow band of frequencies, known as a band gap, regardless of the direction of the propagation. Photons with frequencies outside the band gap propagate through the PBG structure without attenuation. The band gap of the PBG structure can be engineered by varying the lattice periodicity and by varying the design of the unit cell.

By assuming perfect periodicity, we can analyze the performance of a PBG illuminated by a plane wave by invoking the fact that the fields are identical from cell to cell except for a phase shift dictated by the incident angle of the plane wave and the distance between cells. In reality, however, a structure is never infinite in extent, so the assumption of periodicity is always somewhat of an approximation. The effect of truncation is a concern not only to the PBG community, but also to the microwave community.

The PBG structure of greatest interest to us is limited by manufacturing constraints to be a square patch roughly 1 cm by 1 cm. We can put these patches together to cover a large object, but because they are physically so small, this is difficult and leads to departures from periodicity other than truncation. If two neighboring patches are imperfectly butted against each other, they can form a seam. The patches may have to be curved to fit the contour of the object, or they may overlap and again break periodicity. Manufacturing defects may accidentally occur.

All of these breaks in periodicity could be analyzed in a straight-forward manner by putting unknowns over the entire structure. A typical PBG patch is 1000 by 1000 wavelengths, however, which requires approximately 100,000,000 unknowns to solve without invoking any special techniques. Our experience is that for a boundary-element code, 150,000 unknowns overwhelm the computer resources available today so there is quite a disparity between the computational resources needed to solve even a single patch and what is available.

In this project, we explored hybrid computation techniques that exploited the fact that the PBG structures are mostly perfectly periodic and depart from this in local regions. We looked at two problems: the seam between two PBG structures and introduction of a localized defect. Neither problem occurs in the microwave regime, so our study is the first of its kind.

FY 2006 Accomplishments

We applied the Green's function interpolation with a fast Fourier transform (GIFFT) to a complicated structure consisting of a dipole antenna source (acting as a defect) over a metamaterial. The metamaterial consisted of two layers of capacitive loaded split ring resonators (SRR) and was shown to perform like an artificial magnetic conductor. We examined both infinite and finite layers of SRR. This was the first time that the GIFFT technique was applied to metamaterial-type lattices (where the lattice spacing is much less than half a wavelength), so we had to carefully assess the accuracy of the GIFFT approximation.

We demonstrated that GIFFT has significant benefit in matrix fill times over Toeplitz method of moment (MoM) techniques and has solve times that scale as N (number of degrees of freedom) rather than N2. GIFFT also requires less memory than the MoM.

We applied GIFFT to look at the far fields due to a finite array. We discovered that using the current from the infinite periodic solution and truncating it to the footprint of the finite array worked well away from the angular regions around the edge of the array. The field as a function of distance away from the edge as calculated by GIFFT agrees with the diffracted field component predicted by the asymptotic solution of the finite array.

We applied GIFFT to look at the behavior of a seam in a finite array. The seam was formed by viewing half of the array elements as defects and shifting them in a direction parallel to the seam. We learned that the seam had much less of an effect on the far field (even at observation angles near grazing) than the truncation did.

We attempted to incorporate the Green's function for the one-dimensional periodic array of point sources and GIFFT into Sandia's electromagnetic code, EIGER. This is 70 percent complete.

We analytically extracted the diffraction coefficient for two-dimensional seams and made it valid in the plane of the array. This work will be useful in terms of efficiency if we ever take steps to increase the accuracy of the seam calculation by adding fringe currents.

Significance

Numerical modeling of PBG structures in the past has centered on applying a Floquet harmonic expansion of the dielectric function – how the dielectric fills the unit cell. This project applied a boundary element technique that is common in microwave engineering and offers significant advantages in terms of accuracy and efficiency.

The fact that this technique has been applied successfully to solve a PBG structure encourages others in the microwave community to apply microwave techniques to solve PBG problems. Further, although periodic defects in PBG structures have been analyzed, until this work isolated defects have not. Seams have never been analyzed until now.

This project will significantly expand Sandia's ability to model PBG structures in real-world applications and will place us in a position to meet the modeling needs of Sandia, as well as the military and other customers.

Refereed Communications

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F. Capolino, D.R. Wilton, and W.A. Johnson, "Efficient Computation of the 3D Green's Function for the Helmholtz Operator for a Linear Array of Point Sources Using the Ewald Method," to be published in the *Journal of Computational Physics*.

Hydrodynamic Manipulation of Coalescence Dynamics

79771

A. M. Grillet, T. A. Baer, C. J. Bourdon, A. D. Gorby, M. P. de Boer

Project Purpose

Coalescence is a complex dynamic process that determines mechanical properties and stability of emulsions and foams. Because of the wide range of important length scales involved, experimental investigation and development of theoretical models have provided only limited insight into this important phenomenon. Recently, startling new predictions of the effect of external flow on coalescence have suggested that hydrodynamic manipulation of coalescence could be possible. Control of this process has many implications for development of new advanced manufacturing techniques based on two-phase emulsions, where the properties and stability of the emulsion can be affected by processing conditions.

Current research has already shown that emulsion-based processes show great promise to create novel optically active materials, gel structures, and nano-functional particles. By applying hydrodynamic control of coalescence in carefully implemented microfluidic devices, the possibility exists to advance these technologies to new levels of fidelity as well as to open opportunities in sensor development and advanced manufacturing.

In order to access these new application areas, the current understanding of coalescence physics needs to be enhanced. The current model has not yet been validated through detailed experimentation. Due to the complexity of the physical problem, this will require the development of advanced experimental diagnostics that are capable of probing the dynamics of the thin film trapped between the two approaching interfaces during coalescence under controlled external flow conditions. Currently, no theory can explain the coupled effects of surface-active agents and hydrodynamic forces.

Surfactants are known to impact coalescence dynamics due to the Marangoni stresses created at the surface of the drops. An understanding of the coupled surfactant effects is essential for many manufacturing and engineering coalescence applications that contain complex fluids. The resulting validated coalescence model is certain to enhance the predictive capability of our suite of multiphase modeling tools.

FY 2006 Accomplishments

This year we made excellent progress on the individual components, specifically the computer controlled flow cell and the interferometry analysis system.

For the first component, we completed and tested the computer-controlled flow-control algorithm used for tracking the drop position and ensuring an axisymmetric flow. This custom-built system allows precise positioning of the drop within the flow chamber so that it travels along the center line and approaches a repeatable point in the center of the flow chamber. This LabVIEW program required the integration of cameras along the two flow axes as well as flow-control hardware, including valves at the four exit ports of the flow chamber and the syringe pump driving the flow. To test the robustness of the control algorithm, we performed particle imaging velocimetry measurements in the flow chamber.

We made velocity measurements with all of the valves equally open and with the control algorithm operating the valves. Because the resistance of the valves depends on a manually adjusted spring tension, the flow with all valves equally open is not symmetric or balanced. However, we confirmed that with the control algorithm operating, we were able to steer the drop to a repeatable point (within two pixels) of the centerline and under those conditions the flow was well behaved and symmetric.

Concurrently, we developed interferometry analysis capability. For a test system, we used a drop held on the tip of a syringe that was moved toward a stationary glass surface with a linear actuator. This allowed us to generate a dynamic interferogram image series that simulated the type of data that would be collected in our flow chamber. Using that simulated data, we created a robust semiautomated computer program that would analyze the individual interferograms and generate drop shapes. We then correlated those shapes to produce a holistic view of the drop shape over time.

Significance

This project will develop a strategic partnership with Michael Loewenberg of Yale University in order to perform experimental discovery and validation to understand the coalescence process. In the process, we will probe the underlying micro/nanoscale behavior of the thin draining films between approaching fluid interfaces. We believe that coalescence phenomena are omnipresent in processes of interest to engineering sciences, and a fundamental understanding will enhance our ability to provide science-based engineering solutions for our customers.

The experiments exploring thin film drainage under controlled hydrodynamic conditions will provide physical discovery into coalescence dynamics with both clean and surfactant-laden interfaces. This knowledge will provide insight into many processes of interest to Sandia. One example is the advanced manufacturing of nanocoated gelatin. In that project, we used an emulsion to create a dispersion of microspheres and then added a small quantity of surfactant to coat the gelatin and polymerize it to create a hard protective shell.

Without the thin protective shell, the gelatin spheres aggregated and were difficult to use. Unfortunately, since the emulsion was created by vigorously stirring two immiscible fluids, the emulsion contained a wide distribution of drop sizes and potentially nonuniform surfactant coating concentration. By using microfluidic devices that have well-characterized flow rather than a bulk emulsion, this process could be adapted using hydrodynamic control to create very monodisperse spheres encapsulated with advanced functionalized nanocoatings.

Another important outcome of an enhanced physical understanding of coalescence is the development of a validated theoretical model. Because of the range of length scales that are relevant during coalescence events, it is numerically impractical to simulate using finite element methods. Capturing the microscale physics in the thin film region requires high grid resolution and very small time steps. However, to understand processing flows or emulsions that are of practical interest requires the simulation of complex geometries and long processing times.

Currently, approaching interfaces are combined as a numerical convenience. Loewenberg's theoretical model, once validated, would provide important guidelines to model coalescence with physically meaningful time scales that incorporate hydrodynamic and surface effects.

This project will enhance Sandia's technical competency in multiphase flow, which is an important factor in developing science-based engineering of complex manufacturing processes such as encapsulation and foam processing. A better fundamental understanding of coalescence dynamics has many applications, especially in foam manufacture and thermal removal.

Exciting new applications for well-characterized emulsions that have been explored include manufacturing of novel nanostructured and optically active materials, biological materials, microfluidic devices, and microsensors. Fundamental advances in basic science understanding for a process as ubiquitous as coalescence could have impacts not only in engineering sciences, but also in Sandia's homeland security, energy, and infrastructure missions.

Atomic-Scale Modeling of Phonon-Mediated Thermal Transport in Microsystems

79773

S. Aubry, E. S. Piekos, E. B. Webb III, L. M. Phinney

Project Purpose

Thermal management is increasing in importance as the national security mission at Sandia drives the development of micro/nanoscale devices with demanding goals. Limitations in measurement at small scales and high costs associated with design iterations force simulation into an important role in ensuring the success of this mission. Moreover, the decreasing reliability of continuum descriptions of heat transport in microsystems demands accurate atomic-scale modeling. Without this modeling, current Sandia efforts must depend upon unverified assumptions and empirical models representing fundamental interactions of heat carriers with each other and with system features (grain boundaries, surfaces, dopants, and impurities).

To go beyond the existing empirical models, a completely new approach is needed. We propose to build an atomistic model of phonon-mediated thermal transport for subgrid physics input to microsystem device design modeling, generating a robust, multiscale description of thermal transport.

We are using pre-existing and novel molecular dynamics techniques to perform simulations explicitly examining phonon-microstructure and phonon-phonon interactions across a range of temperatures, providing a detailed characterization of phonon transport at the atomic scale. We will analyze the simulation results to produce subgrid models of phonon transport for use in Sandia's mesoscale models which, in turn, provide input to device-scale models. Comparison against existing experimental data and further measurements of thermal conductivity over a broader range of temperatures in well-characterized polycrystalline samples will validate the new models.

Starting from atomic-scale simulations, models of phonon transport at all length scales will be enhanced. The benefit will be more accurate predictions of the lifetime and reliability of microscale devices in essential systems. Designers will also benefit by avoiding costly and time-consuming design iterations, and they will be able to explore regimes far outside current practice with confidence.

FY 2006 Accomplishments

We used molecular dynamics simulations to extensively characterize harmonic phonon-grain boundary scattering at the atomic scale. We completely characterized scattering off a high-energy grain boundary at the atomic sale. Our results indicate that scattering depends mainly on the frequency of the incident phonon. Low frequencies are scattered least, while high frequencies are scattered more strongly. We used this atomic-scale data to compute the Kapitza conductance of the grain boundary to yield a prediction that may, in principle, be compared directly against experiment.

We find that the aggregate effect of all phonons interacting harmonically with a high-energy grain boundary is well approximated by a simple model whereby each phonon is predicted to have an equal probability of transmission or reflection at the grain boundary. We also examined impurity effects on scattering at the grain boundary. We find that a large impurity concentration at the grain boundary is required in order to produce a large change in phonon-grain boundary interactions.

We incorporated this data into a mesoscale code, completing an initial model of phonon-grain boundary scattering. Direct simulation Monte Carlo results using this initial model indicate that low-temperature thermal conductivities are over-predicted and highlight the need for precise grain structure characterization for precise comparisons between predictions and experiment.

We are investigating anharmonic scattering effects at nonzero temperature using molecular dynamics, and the completion of this work will allow for more refined mesoscale models.

We carried out thermal conductivity measurements of redesigned test structures, and these measurements are being supplemented with measurements using an additional cryostat to extend the temperature range of the data. The measurements of thermal conductivities for the different structural layers of the Sandia's SUMMiT V™ (Sandia ultraplanar, multilevel MEMS technology 5) process indicate that the thermal conductivity is a function of the particular SUMMiT layer. At low temperatures, the variation in thermal conductivity of the differing layers can be quite complicated. As the temperature increases above room temperature, the difference in thermal conductivity between the layers decreases. A simulation of a test structure indicates that bond pad heating likely contributes to the measured thermal conductivities being up to 10 percent lower than actual values.

The data collected so far provide thermal conductivity data as a function of temperature for Sandia's SUMMiT layers for device simulations and model validation; however, the measured data is insufficient to fully explain all of the observed trends. In order to validate first principles thermal conductivity models, an ideal data set would be for a range of polycrystalline silicon microstructures and doping values. Also, the polysilicon films should be well characterized so that the distribution of grain sizes and orientations are available to the modeling efforts. Next year we will work to acquire thermal conductivity measurements for well-characterized polysilicon test structures with various microstructures and doping.

Significance

Our atomic-scale molecular dynamics simulations enabled us to provide the most complete picture to date of the dependence of phonon-grain boundary scattering on frequency, wavelength, and angle of incidence. We used these results to compute the value of an experimentally accessible quantity, and this data is sufficient to construct a mesoscale model for phonon-grain boundary interactions. With this work, we introduced two new molecular dynamics techniques for wave packet simulations that allow us to increase the efficiency of the method by simulating many phonons simultaneously and modeling arbitrary angles of incidence.

As we use molecular dynamics simulation to make increasing contact with experiment, our results become of interest to the greater academic community. The utility of these results for mesoscale modeling will enhance Sandia's ability to use simulations to aid in the microsystems design process. This enhancement is furthered by recent developments in our direct simulation Monte Carlo mesoscale modeling effort to incorporate the effects of grain structure into a Monte Carlo simulation.

Our thermal conductivity measurements of the SUMMiT V test structures provided useful data that will support first principles and mesoscale modeling efforts at Sandia. This data will also provide the accurate thermophysical properties needed to improve our current modeling efforts for microscale devices. The experimental work also extended the temperature range of existing data for Sandia's SUMMiT V process and was the first effort to characterize all five of its polysilicon structural layers. These efforts will provide crucial data and vital models to support Sandia's microsystems customers and its stockpile stewardship and national security missions.

Refereed Communications

C.J. Kimmer, S. Aubry, A. Skye, and P. Schelling, "Scattering of Phonons from a High-Energy Grain Boundary in Silicon: Dependence on Angle of Incidence," to be published in *Physical Review B*.

Multiphase Dynamics of Soft Biological Tissues

79774

R. E. Jones, B. L. Boyce, T. D. Nguyen

Project Purpose

Biological tissues are uniquely structured materials with technologically appealing properties. Soft tissues, such as skin, are constructed from a composite of strong fibrils and fluid-like matrix components. This is the first coordinated experimental/modeling project at Sandia and in the open literature to consider the mechanics of fibril-based anisotropy, viscoelasticity, and the coupled solid-fluid deformation of soft biological tissues. We are exploiting and applying Sandia's expertise in micromechanical experimentation and continuum mechanics modeling to soft materials of biological origin.

This project's purpose is to provide a detailed understanding of the deformation of ocular tissues, specifically the highly structured skin-like tissue in the cornea. This discovery will improve our knowledge of soft/complex materials testing and modeling, our ability to design new biomedical microelectromechanical systems prosthetics, and our understanding of collateral damage caused by munitions. Furthermore, a clear understanding of the mechanics of soft tissues will lead to bioinspired materials or applications of natural materials, such as highly supple and impact resistant body armor. The models developed in the project will also be applicable to modeling the deformation cells for cell manipulation and disease/infection identification.

FY 2006 Accomplishments

In our second year we successfully devised preconditioning treatments and statistical analysis to enable meaningful material response characterization of excised tensile strips. We compared this data to a quasilinear viscoelastic extension of the state-of-the-art published model for the cornea and found the model lacking. We developed a new model that was able to capture the observed stress-dependent viscoelastic behavior and showed it to be predictive. Our predictive model has numerous applications in biomedical design.

Significance

This project has external and internal significance. Within Sandia, the DOE complex, and the Department of Defense, there is a pressing need to create better body armor and to be able to predict collateral damage due to munitions. The foundation for solving these problems is the understanding and the predictive material model we are developing.

Outside the laboratory, the potential applications of this work to soft tissues and specifically to surgery and diseases of the cornea are illustrated by a number of recent National Institutes of Health (NIH) calls. Our plans include pursuing NIH funding in order to bring the impact of our research to medical practice.

Lastly, given the unique toughness and suppleness of soft, hydrated biological tissues, we believe there are numerous biomimetic opportunities for design applications of our work.

Refereed Communications

T.D. Nguyen, R.E. Jones, and B.L. Boyce, "A Non-linear Viscoelastic Model for the Tensile Behavior of Bovine Cornea," in *Proceedings of the ASME 2006 Summer Bioengineering Conference*, p. 1, June 2006.

Other Communications

B.L. Boyce, T. Nguyen, R. Jones, J.M. Grazier, R. Regueiro, and R.K. Nalla, "In Vitro Anisotropic Viscoelastic Properties of Bovine Cornea Tissue," presented at Material Research Society Fall meeting, San Francisco, CA, August 2005.

T.D. Nguyen, R.E. Jones, B.L. Boyce, J.M. Grazier, and S. McLeod, "Characterization and Modeling the Mechanical Behavior of Bovine Cornea," presented at 43rd Technical Meeting of the Society of Engineering Sciences, State College, PA, August 2006.

Multilength Scale Algorithms for Failure Modeling in Solid Mechanics

93525

J. D. Hales, M. W. Heinstein, J. R. Overfelt, A. B. Williams, H. C. Edwards, J. E. Bishop, A. S. Gullerud, J. T. Ostien

Project Purpose

The class of problems we need to solve are those where structural models cannot be readily refined (e.g., h-adaptivity) to a length scale appropriate for failure prediction.

Specifically, features such as welds and bolted connections in these structural models are typically omitted, yet they are the likely locations where failure initiates. It is important to understand that it is necessary to make such omissions so that the structural scale model can reasonably be solved in an explicit transient dynamics setting. Incorporation of fine-scale features would otherwise dramatically increase the cost of the calculation – not because of the increased size of the model but because of the corresponding decrease in the stable time-step needed to integrate the equations of motion forward in time.

We propose to solve this problem with:

- a computational multiscale methodology for bridging the structural model (reference-length scale) to continuum model (fine-length scale) where multiple length and time scales are active
- multiple discretization methods for macroscale failure response developed with rapid algorithm prototyping within the computational mechanics framework SIERRA.

FY 2006 Accomplishments

This past year, we initiated development of a multi-scale capability in the SIERRA framework. We are demonstrating the ability to couple Presto and Adagio for multiscale analysis. A multipoint constraint for the multiscale boundary condition coupling is in place, as is a volume-averaging multiscale homogenization volume constraint.

Time-stepping flexibility is being expanded to support the determination of the accuracy of explicit-implicit coupling. And we discovered an area that requires further research, namely the homogenization of material moduli.

Significance

Our work this year is a step foward in the ability to model structural failure in a computationally efficient way. By solving a detailed submodel in an implicit or quasistatic manner, we can allow a larger structural model to run without the penalty of a very small time step. The submodel will have the fidelity required to capture failure, and that will be reflected in the overall structural response.

The technology is applicable where failure modeling is important. Examples include welded or bolted joints in any number of structures, crack propagation, and localized buckling. This technology is also relevant when information about a component is required when that component is driven by the overall structural response. In this instance, the submodel may be analyzed to determine its normal operating response and not its failure characteristics.

We demonstrated that there are limits to the number of explicit steps that can be taken in the system model before an implicit or quasistatic step is taken in the submodel. More experience is needed here. We also showed that the multipoint constraint approach to the boundary condition coupling is simple, but this is known to be an inaccurate approach. Furthermore, we demonstrated that the volume constraint needs further investigation to be applicable to a wide range of problems.

Homeland Security

Universal Biosample Processor

79812

B. A. Simmons, R. V. Davalos, K. L. Krafcik, A. M. Morales

Project Purpose

The goal of this project is to create a universal biosample processor system that processes biological samples in complex matrices. Efficient and reliable particle separators and concentrators are needed to support a wide range of analytical functions such as pathogen detection, sample preparation, high-throughput particle sorting, and biomedical diagnostics.

The advent of lab-on-a-chip devices based on the phenomenon of dielectrophoresis offers several advantages for meeting some of the challenges associated with cell sorting and detection. The majority of the devices presented in the scientific literature have used glass-based devices for these applications, but recent activity indicates that polymer-based devices are as effective as their glass progenitors.

Processing and operational advantages motivate the transition from glass and silicon to polymer microdevices, specifically the availability of numerous innate chemical polymer compositions for tailoring performance, mechanical robustness, economy of scale, and ease of thermoforming and mass manufacturing. Polymer-based microfluidic devices have been developed and used for liquid/liquid and particle separation and other lab-on-a-chip applications, including capillary electrophoresis, miniaturized polymerase chain reaction chambers, nucleic acid analysis, protein analysis, and fluidic mixers.

The main appeal of these polymeric devices is that they are relatively inexpensive and produced employing standard mass fabrication techniques such as injection molding and hot embossing instead of the costly per wafer technique of microlithography. We have made significant progress toward, and produced significant results using, polymeric dielectrophoretic devices for selective trapping, concentration, and gated release of a range of biological organisms and particles.

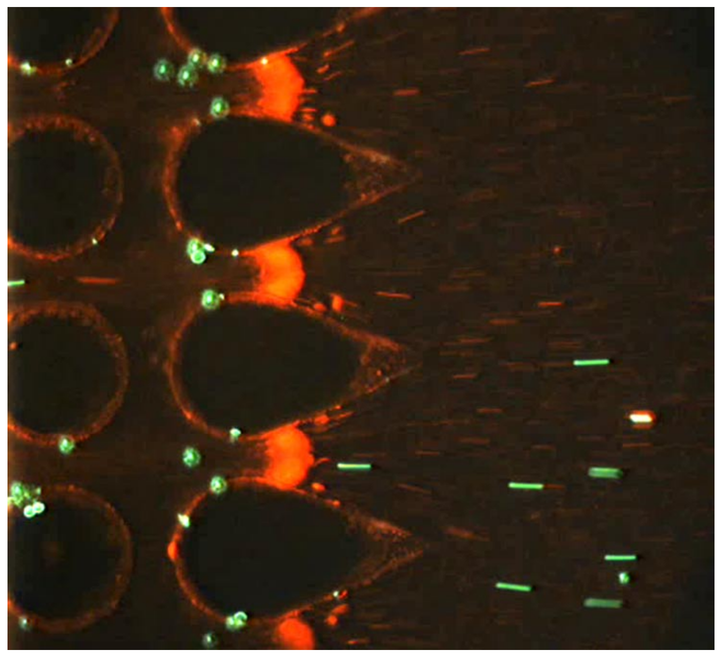

Image from a digital movie showing the differentiation between spores (red) and beads (green). Posts are 0.2 mm in diameter.

FY 2006 Accomplishments

We successfully completed the transition to polymer-based devices. While glass-based insulator-based dielectrophoresis (iDEP) microdevices perform well, sample throughput is generally low because of the geometrical limitations present in isotropically etched devices. Typical sample flow rates for glass-based devices are in the range of ten microliters per hour. In contrast, polymer-based iDEP devices can be easily scaled to handle much larger sample volumes using commercially available and inexpensive techniques that produce much deeper features and larger channel volumes.

Our group made polymeric iDEP elements from cyclic olefin copolymers, such as Zeonor®. Cyclic olefin copolymers have received a significant amount of recent interest in microfluidics owing to their low auto-fluorescence and high chemical resistance to a wide range of polar solvents; such properties support the use of this class of polymer for iDEP devices. We demonstrated the capabilities of polymer-based DEP and iDEP devices to separate and concentrate waterborne bacteria, spores, and particles.

Significance

Particle sorting and separation is a vital aspect in the performance of numerous lab-on-a-chip applications and will remain so for the foreseeable future. Our work indicates polymer-based DEP and iDEP devices are effective for the selective trapping and concentration of a wide range of particles. The nonuniform electric field required to generate DEP can be generated by a variety of intrinsic structures and design methodologies. These include arrays of metallic electrodes contained within a polymer microfluidic device (DEP), or by applying a field across a polymeric microchannel containing posts, ridges, or other insulating obstacles (iDEP). Regions of high field intensity generated between these insulating posts repelled insulating particles dielectrophoretically by various degrees, producing selective and field-tunable particle traps.

The performance of the polymer-based DEP microdevices at removing and concentrating cellular and subcellular particles selectively is similar to that obtained in the glass-based DEP microdevices. These results illustrate the great potential of polymer-based DEP and iDEP devices for use in the concentration and sorting of a wide variety of cells, particles, proteins, and DNA. We envision a role for these polymeric devices as cost-effective and disposable tools used in multiple front-end sample preparation applications that will enhance current detection techniques.

Refereed Communications:

B. Simmons, G. McGraw, R. Davalos, G. Fiechtner, Y. Fintschenko, and E. Cummings, "The Development of Polymeric Devices as Dielectrophoretic Separators and Concentrators," *MRS Bulletin*, vol. 31(2), pp. 120-124, February 2006.

R. Davalos, A. Morales, K. Krafcik, and B. Simmons, "Dynamic Surfactant Coatings Improve Performance of Dielectrophoretic Devices," submitted to *Lab on a Chip*.

Other Communications

B. Simmons, "Polymer-Based Dielectrophoresis," presented at the MRS National Spring Meeting, San Francisco, CA, 2006.

Security-Enabled Programmable Switch for Protection of Distributed Internetworked Computers

79813

J. Van Randwyk, L. G. Pierson, P. J. Robertson, E. L. Witzke, B. R. Hamlet, N. A. Durgin, J. R. Hamlet, B. D. Kucera

Project Purpose

Increasing computer security concerns make it desirable to push security closer to the desktop; however, it is not practical or feasible to place security and monitoring software on all computing devices. We are creating a hardware and software architecture that will enforce security policies by pushing security functions closer to the end user without interfering with desktop environments. To that end, we are developing a specialized programmable Ethernet network switch.

Embodied in this device is the ability to detect and mitigate network attacks that would otherwise disable or compromise the end user's computing nodes. We call this device a secure programmable switch (SPS). The SPS is designed with the ability to be securely reprogrammed in real time to counter rapidly evolving threats, such as fast moving worms. The ability to remotely update the functionality of the SPS protection device is cryptographically protected from subversion.

With our concept, the user cannot turn off or fail to update virus scanning and personal firewall filtering in the SPS device as he/she could if implemented on the end host. The SPS concept also provides protection to simple/dumb devices such as printers, scanners, and legacy hardware.

FY 2006 Accomplishments

After investigating numerous security functions to be provided by the SPS, we chose to study the detection and mitigation of distributed denial of service attacks. We then designed and built a small network test bed in which to demonstrate successful detection and mitigation of such attacks. We developed the code for the detection and mitigation of this initial attack scenario based on an intrusion detection system developed at Sandia called MON, and we exercised these components in the isolated network test bed.

We applied previously developed concepts in the cryptographic assurance of execution correctness to the chosen embedded hardware platform and compiled an Altera NIOS-II "soft core" central processing unit (CPU) into a field-programmable gate array (FPGA) along with the cryptographic means to decrypt and authenticate the protected instruction stream. We examined methods for encrypting the compiled hardware to protect the physical security of the reconfigurable logic to be loaded into the prototype platform. This encryption of the FPGA hardware configuration is separate from the cryptographic software protection described above.

We developed the ability to shrink-wrap a program intended to be protected by encrypting and signing the instruction sequence and its data, heap, and stack areas. In an FPGA we prototyped the ability to unwrap such a protected program by decrypting and authenticating the instruction sequence within the CPU. This provided the ability to protect not only simple programs, but also more complex programs that manipulate the stack and the heap as well as simple static data structures.

After applying this technique to protect programs that extensively used interrupt processing, we found that the protected instruction fetch rate was insufficient to perform both the interrupt processing and the main program processing. This necessitated the acceleration of the protected instruction fetch process with specialized hardware, including the decryption and authentication of the instruction stream.

This need to deal with the cryptographic overhead of the slow prototype prompted us to redesign the implementation of the secure switching function and the development of hardware acceleration of the cryptographic functions. While this change prevented full exploration of the use of the prototyped SPS in a network test bed environment before the end of the project, the hardware acceleration now enables the prototyping of the protection of larger, more complex and wider variety of programs as they execute.

Significance

An SPS distributed network protection device will allow finer-grained control of security functions and enable cooperative network monitoring resulting in rapid detection of new, undetected threats. This device will enable monitoring for insider and outsider threats, necessary components of a robust security architecture. Because this device would sit in line with a user's network connection, and presumably be required for network access, end-user devices without built-in security capabilities, such as printers or personal digital assistants, would be protected from malicious traffic.

This project demonstrated the basic feasibility of the SPS. In addition, the enhancements to the underlying protection technology now enable protection of large, complex embedded programs from subversion, thereby leveraging the security of other high-assurance applications of interest to homeland security and information operation program areas.

Referred Communications

L.G. Pierson, P.J. Robertson, T.J. Toole, and J. Van Randwyk, "Protection of Distributed Internetworked Computers," in *Proceedings of the 2005 IEEE International Carnahan Conference on Security Technology*, pp. 212-215, October 2005.

Automated Terrorist Threat Detection System

79815

J. J. Carlson, W. A. Amai, T. H. Ko

Project Purpose

The purpose of this project was to develop automated video screening technology to assist security forces in protecting our homeland against terrorist threats (e.g., the covert placement of bombs inside crowded public facilities).

Although video-surveillance systems are increasingly common, current systems cannot detect the placement of bombs. It is also unlikely that security personnel could detect a bomb or its placement by observing video from surveillance cameras. The problems lie in the large number of cameras needed to monitor large areas, the limited number of security personnel employed to protect these areas, and the intense diligence necessary to effectively screen live video from even a single camera.

Different from existing video-detection systems designed to operate in nearly static environments, we are developing technology to detect changes in the background of dynamic environments – environments where motion and human activities are persistent over long periods. Our goal is to quickly detect background changes, even if the background is visible to the camera less than five percent of the time and possibly never free from foreground activity. Our approach employs statistical scene models based on mixture densities.

We hypothesized that the background component of the mixture has a small variance compared to foreground components. Experiments demonstrate this hypothesis is true under a wide variety of operating conditions. A major focus involves the development of robust background estimation techniques that exploit this property. We desire estimation algorithms that can rapidly produce accurate background estimates and detection algorithms that can reliably detect background changes with minimal nuisance alarms.

Another goal is to recognize unusual activities or foreground conditions that could signal an attack (e.g., large numbers of running people, people falling to the floor, and so on). Detection of background changes and/or unusual foreground activity is used to alert security forces to the presence and location of potential threats.

FY 2006 Accomplishments

We made significant technical strides. We sped up the background estimation process in the presence of extreme foreground activity. We examined and implemented several techniques to reduce nuisance alarms in our detection algorithms. We continued a collaborative effort with security personnel at the Albuquerque International Sunport to both test and evaluate our technical developments. We mounted four cameras at the airport (two interior and two exterior) and collected data from each camera for algorithm development, testing, and evaluation.

Our research partners at New Mexico State University's Physical Science Laboratory investigated traditional approaches (e.g., Pearson's method of moments) for estimating the parameters of a mixture density. Although traditional methods are generally computationally intensive, our goal was to see whether the known mixture characteristics in the background-monitoring problem could be exploited to reduce the computational requirements. We compared theperformance (estimation accuracy, robustness, computational requirements, speed, and so on) of Pearson's method of moments to the methods we developed and found that although Pearson's method of moments can produce background estimates more quickly, it is not as statistically robust as ours.

Another focus of our work was to integrate foreground-monitoring capabilities, including the capability to detect unusual foreground activities. We also investigated how multiple cameras monitoring a common area can be used to speed up the background estimation process. We installed several systems at the Sunport for extended development, testing, and evaluation.

Significance

The development of enabling technologies is among the highest priorities of both Sandia's Homeland Security and Defense Strategic Management Unit and the National Strategy for Homeland Security. The technology we developed has the potential to dramatically impact the ability of our already overburdened security forces to protect our citizens from terrorist attacks. This capability will provide key advantages and has tremendous potential for both detecting and preempting terrorist acts.

The successful application of our research developments will significantly strengthen our nation's ability to fight terrorism. In addition to homeland security initiatives, our work could also impact several other Sandia initiatives, including physical security, intelligence, and military technology and applications.

The key goal of our project was to develop robust, cost-effective, and practical video screening technology that effectively assists security personnel in protecting our homeland against terrorist threats and that can be quickly commercialized. Major technical goals included both detecting background changes when background is observable less than five percent of the time and detecting changes quickly enough to give security personnel time to respond to and, hopefully, to preempt an attack.

Another key goal was to demonstrate the benefits of using three-dimensional techniques in conjunction with the new background change detection algorithms. Yet another goal was to combine both background and foreground monitoring capabilities into one integrated system.

We successfully demonstrated the achievement of each of these goals in a realistic facility-monitoring application. As a result, we have developed several proposals that are intended to take us beyond the applied research phase of development provided by this project. We are pursuing collaborations with both universities and industry.

Other Communications

J.J. Carlson, C.A. Jenkins, and J.B. Jordan, "Background Change Detection in Video Using Robust Parameter Estimation Techniques," to be published in *SPIE Conference*.

Development of Ultraminiaturized Photomultiplier Detectors

79818

M. Malinowski, C. P. Yang, J. T. Hachman Jr., C. A. Steinhaus

Project Purpose

The primary goal of this project was to develop miniaturized photomultiplier tubes (mPMTs) made by LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) fabrication technology.

These mPMTs would incorporate the unique characteristics of structures made by the LIGA process: These structures could have high aspect ratios, would be metallic (up to ~ 1 mm high), and would be effectively extrusions of two-dimensional (2D) patterns because of the normal x-ray incidence used in PMMA (polymethyl methacrylate) mold exposures. An insulating substrate, such as 1 mm thick alumina, would be used as the base for the mPMT structure. The prototype mPMT made by the LIGA process would be ~ 10 mm x 10 mm x 4 mm thick.

A PMT is based on an electron multiplier structure that multiplies the electron signal created by electrons emitted from a photocathode located at the multiplier input. Multiplication occurs by repeated impact on secondary emissive surfaces, called dynodes, arranged such that the electrons emitted from one dynode or dynode row impact on a successive dynode or dynode row. The output from the last dynode or dynode row hits a collector electrode.

In the current concept, the LIGA process would be used to make the metallic structures in the electron multiplier structure. The geometry of the mPMT electrodes and their numbers, and geometrical relationships between dynode elements, can be varied and optimized by the relatively straightforward process of making new x-ray mask sets. The same LIGA processing can be used to make a variety of different mPMT structures.

In most LIGA applications, the electroplated structures are usually separated from the substrate. However, the electron multiplier structure that forms the heart of the LIGA mPMT to be made would not be separated from the substrate but would be electroplated onto conducting electrode lines patterned atop an insulating substrate. The entire assembly-plated electrode parts, electrical contact lines, and insulating substrate would form a unit assembly, an mPMT chip. A potential difference of approximately +100 V would be applied between each dynode row and between the last row and the electron collector structure.

The 2D, extrusion-like nature of the mPMT electrode elements and the alignment/connection of the electrode elements to the electrode contact lines are unique to the mPMT developed in this project. Although LIGA has been used in the past to make

Figure 1. Bonded mPMT chips (left - Venetian blind; right - single channel)

single, electron multiplier dynode stages, these stages required assembly after LIGA synthesis, a conventional fabrication technique.

In contrast, our novel LIGA mPMT fabrication technique uses the x-ray exposure of an aligned x-ray mask, resulting in a LIGA mold where the mPMT structural elements are automatically aligned and connected to the contact lines. Subsequent electroplating into the mold results in mPMT structural elements that are effectively "assembled" onto electrical contact lines – no additional assembly is necessary.

FY 2006 Accomplishments

- Refined the "Venetian blind" mPMT design based on lessons learned in FY 2005.
- Made the structures in the original Venetian blind design, which were fragile and had low yield in processing, more robust in order to improve structural strength. This change resulted in higher fabrication yields in FY 2006.
- Designed an additional mPMT – the focused, single channel mPMT.
- Performed 2D electron optics modeling to make a compact, mechanically robust structure. The simplicity of this single channel mPMT compared to the Venetian blind could eventually provide a fabrication advantage.
- Designed and made a new mask set containing the refined Venetian blind and single channel mPMTs.
- Developed and proved a complete LIGA processing sequence for making nickel mPMT structures on alumina substrates.
- Performed four wafer x-ray exposures using a new mask set containing the refined Venetian blind and new single channel designs. Of the four wafers processed, one had mPMT structures ~ 0.6 mm high and had ~ 85% yield; a second wafer had ~ 0.9 mm high structures and had ~ 25% yield. Two wafers were scrapped because of processing issues.
- Mounted and wire-bonded mPMT chips (10 mm x 8 mm) onto holders.
- Made electrical connections to the mPMT elements by two different techniques based on gold wire bonding. Lead resistances could be made as low as about 2 ohms between each mPMT electrical element and the corresponding lead in the dual inline package containing the mPMT.
- Successfully performed high-voltage testing of a single channel mPMT.
- Used a commercial high-voltage (HIPOT) tester verify high-voltage standoff between all relevant mPMT elements while under vacuum. Alternating current voltages used in this testing were equivalent to greater 1.5 times the expected direct current voltages necessary for device operation.
- Dynode coating materials (MgO and CsI) were successfully deposited onto test coupons.
- Electrophoretic deposition of magnesium oxide (MgO) on test coupons was successful, with secondary electron yields (SEYs) of ~ 1.6 measured at ~ 100 volts with a direct current electron beam; higher yields are expected for MgO deposited on mPMT dynodes because of the high-electric fields present in the interdynode spaces. Cesium iodide (CsI) was also deposited on test coupons and found to have a SEY of ~ 3.4 at 150 V; this yield was also measured with a direct current electron beam.

Significance

The mPMT work done in the first two years of this project is significant in its demonstration of the ability of LIGA to produce a high-voltage electrical device for use in vacuum. A unique combination of aligned x-ray exposure, plating base composition (TiO_x/Au/Cr), substrate (alumina), and contact line wire bonding methods resulted in the production of miniature, electron multiplier structures that were effectively self-assembled on alumina substrates by the normal plating step in the LIGA process. All materials in the mPMT were chosen to be ultrahigh vacuum compatible in order to facilitate possible future integration of the mPMT into a vacuum package.

The development and refinement of the precise LIGA processing steps were crucial to the ability to produce these first mPMT structures having structural integrity and the necessary high-voltage standoff characteristics. The results of this work could have application in development of other miniature vacuum devices, a unique capability that could have application in Sandia's mission areas. For example, portable detection of airborne chemical agents and nuclear radiation requires amplification of detected signals.

In these detection systems, signal amplification of light can be performed by a variety of amplification devices including PMTs. PMTs have been made smaller by different manufacturers in order to help meet the size reductions in detection systems dictated by portability. Although small PMTs exist, complete, successful development of the current mPMT could lead to a new, smaller species of PMTs.

Portable Medical Diagnostic System for Detection of Presymptomatic Biomarkers of Chem/Bioagent Exposure

79820
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Project Purpose

Our goal is to develop a portable device for rapid screening and triaging of a population after a suspected incident of bioterrorism. The device will carry out multianalyte detection rapidly (in minutes) using a tiny amount of blood or, in the future, saliva. The presymptomatic detection will be based on detection of early-response immune biomarkers, such as cytokines, and growth factors that are produced within minutes to hours after infection.

Detection of a single biomarker is not useful, but quantitative detection of multiple biomarkers may provide a characteristic signature for a pathogen. The device will also be capable of detecting more definitive and specific markers, such as surface antigens and toxins, or late immune response markers such as antibodies.

The proposed device will be based on microfluidic technologies to manipulate and concentrate biomolecules and to perform selective recognition of analytes using immunoassays. Multiple analytes will be detected using receptor arrays fabricated in microchannels. The diagnostic assay will be performed in a glass or plastic chip. Blood can be introduced into the chip with minimal sample preparation. The cells will be filtered out or concentrated using dielectrophoretic traps that use simple-to-design and inexpensive-to-fabricate faceted structures in a channel.

Biomarkers present in serum or in immune cells (macrophages, T cells) will be analyzed using microarrays of immobilized receptors (scFv, antibodies, or synthetic ligands). The existing recognition-based assays suffer from extended time (hours) required for diffusion-limited binding. Recently discovered electrokinetic methods for recirculation and concentration will enable binding reactions to be completed in minutes. Fluorescently labeled secondary antibodies will be used to transduce binding into a signal. Inexpensive red diode lasers will provide the excitation source, and fluorescence will be detected using diode array.

FY 2006 Accomplishments

Using ridges in microchannels, we trapped (parallel ridges perpendicular to fluid flow, applied field 20 V/mm) and deflected (parallel ridges, angled to the fluid flow, applied field (30 V/mm)) macrophages in a very reproducible manner. We concentrated cells using ridges perpendicular to the fluid flow to make them travel “single-file” in a line across a channel and then collected them in a specific region of a channel using a ridge angled to the flow.

We demonstrated the ability to use electrokinetic phenomena for mixing. When a hydrodynamic flow is present in opposition to the electrokinetic flow, recirculation of the particles occurs before each of the whole ridges (ridges that transverse entire channel). The extent of mixing and mixing time can be controlled by the number of ridges present. We found that when ridges do not completely transverse the entire channel, recirculation also occurs at the end of the ridges. This newly discovered mixing scheme will be used to mix lysed or permeabilized macrophages and T cells with antibodies to significantly reduce the time required for an immunoassay (less than two minutes).

We also made significant progress in our goal of biomarker discovery. We exposed macrophages and dendritic cells to anthrax lethal toxin and monitored cytokine expression. We saw a clear reduction in cytokine (IL-2, IL-4, IL-6, IL-10, IL-13, TNFa) production in cells exposed to lethal toxin. We developed microchip immunoassays for Il-6 and TNFa; immunoassays for additional cytokines are under development. The microchip immunoassays we developed are fast (minutes), sensitive (limit of detection of pM), and can be multiplexed.

Significance

National security is a key mission at Sandia, and this project represents a major advance in the area of diagnosing (and hence protecting) the US population in the event of a bioterrorism event. This device could be extended to other point-of-care applications such as cancer diagnosis, emerging infectious diseases (e.g., severe acute respiratory syndrome or SARS), and foot-and-mouth disease in cattle, allowing Sandia to impact health care and commerce in a significant way.

Sandia has been a key player in development of portable devices for detection of bioagents in environmental samples such as water or aerosol. This device fills a need not previously targeted – rapidly screening people potentially exposed to a bioagent. Efforts such as this will allow us to develop partnerships with the National Institutes of Health (especially the National Institute of Allergy and Infectious Diseases), complementing our strong partnerships with Department of Homeland Security, the Department of Defense, and industry, to establish a comprehensive program in biodefense.

Refereed Communications

L.M. Barrett, A.J. Skulan, A.K. Singh, E.B. Cummings, and G.J. Fiechtner, "Insulator-Based Ridges for the Manipulation of Particles and Cells in Microchannels," in *Proceedings of the MicroTotal Analytical Systems*, p. 1, November 2005.

L.M. Barrett, A.J. Skulan, A.K. Singh, E.B. Cummings, and G.J. Fiechtner, "Dielectrophoretic Manipulation of Particles and Cells Using Insulating Ridges in Faceted Prism Microchannels," *Analytical Chemistry*, vol. 77, pp. 6798-6804, November 2005.

Cognitive Modeling of Human Behaviors

93565
M. A. Ehlen, D. K. Belasich, R. A. Allen, C. J. Gieseler, C. Lynn, A. J. Scholand, N. M. Berry, E. D. Eidson, J. C. Forsythe, M. L. Bernard

Project Purpose

Of the six metrics the Department of Homeland Security (DHS) uses to measure the impacts of terrorist attacks, public confidence and economic impact are arguably the most intertwined. To maintain national security, the US needs a healthy and productive workforce, and the nation's sense of well-being requires individual and collective economic stability. However, the majority of modeling and simulation that supports DHS public policy considers these two metrics independently.

To understand and provide true economic security, DHS public policy needs integrated modeling of the traditional factors that drive this economic "engine" and the affective social and cultural factors that influence people's participation in this engine. Our goal is to develop a new cognitive science-based modeling framework of the individual-level decision making that is critical to national economic security.

In addition to baseline models of economic/cognitive modeling, research is required in two areas specific to extreme events: first, in events such as terrorist attacks, the cognitive and affective portions of brain likely coordinate differently than during normal events. We need to understand how actual brain function and decision processes differ during extreme events. Second, an extreme event creates a situation where the communication between people of their interpretation of the event can dominate an individual person's own interpretation of the event, that is, "group think" can dominate "individual think." We need to understand how social communication and cognition affect overall economic decision-making.

By expanding the current state-of-the-art cognitive systems modeling and economics, and simulating them in large scale, Sandia will be able to provide realistic, high-fidelity, and theoretically consistent analysis of the impacts of acts of terrorism on the economy and public confidence.

FY 2006 Accomplishments

Conducted Survey of Cognitive Science Economic Decision Making Research

We conducted an extensive literature review and combined it with the existing Sandia cognitive modeling framework (CMF) to formulate an economic cognitive modeling framework. We reviewed 60 articles from two main areas of research: 1) neuroeconomics, a new discipline that uses neuroscience, economics, and psychology to study how the brain evaluates decisions, categorizes risks and rewards, and interacts with others; and 2) behavioral economics, which applies scientific research on human and social cognitive and emotional biases to better understand economic decisions and how they affect market prices, returns, and the allocation of resources.

Identified Modeling and Simulation Scenario

To drive development of the software implementation of this economic CMF, we selected an analysis scenario on the economic impacts of a pandemic influenza, a subject currently of high priority to DHS.

A full-blown national pandemic fits our project's description of an extreme event: it could cause significant death (upwards of two million people), national economic impacts in the hundreds of billions of dollars, and significant losses to public confidence and economic demand, the latter of which is often the largest uncontrollable shock to an economy. A full-blown pandemic allows us to examine the complex set of emotive and rational processes people must conduct due to the uncertainties associated with if, when, and at what levels people will become sick and die.

Began Development of Economic Cognitive Modeling Framework (E-CMF)
While this task was originally scheduled for FY 2007, we found it necessary to start developing the software architecture in FY 2006 to ensure that we have a cognitive/economic model framework that is both sufficient in its detail to adequately model cognitive processes and that is scalable to potentially millions of agents. The development of the E-CMF proceeded along two parallel tracks: a C++ implementation in N-ABLE and a Java implementation, where both models use the same CMF "paper model" specification. Based on this work, we decided to proceed in FY 2007 with only the C++ implementation so that we can fully parallelize the code and implement it in N-ABLE.

Significance

Defense Against Terrorism
Unlike most pre-9/11 threats to the US in which the government is the primary responder, terrorist attacks demand readiness and response from all sectors and classes of people. This modeling and simulation project will provide a unique Sandia capability that could be used by DHS to model and assess how the populace within the economy responds – and could best respond – to acts of terrorism, and how to keep the underlying economy stable and resilient.

Protecting our National Infrastructures
Humans physically operate the entire sociotechnical, economy-infrastructure system. Understanding how they experience and respond to attacks to the economy and underlying infrastructure will result in more science-based, realistic, and effective critical infrastructure protection policy.

Providing New Capabilities to our Armed Forces
New socioeconomic modeling capabilities could significantly improve the design and efficacy of economic, social, and cultural recovery plans for a foreign nation before and after US invasion.

Massive Graph Visualization

93566
K. D. Moreland, B. A. Hendrickson, A. T. Wilson

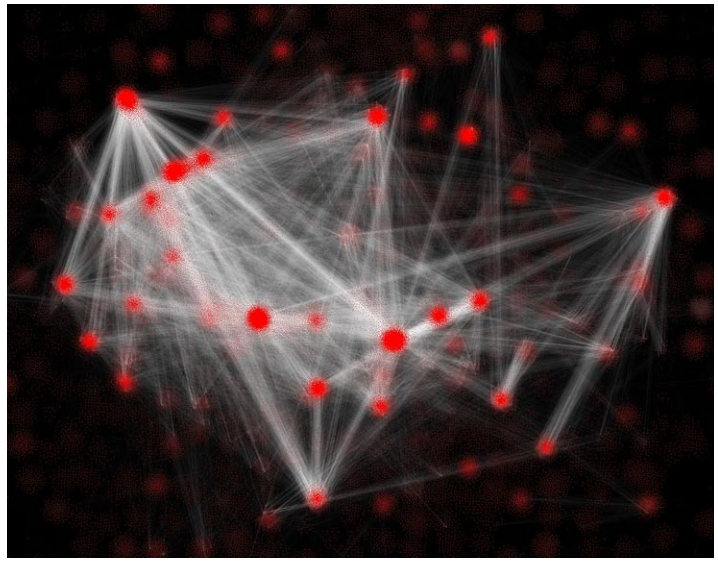
Project Purpose

The purpose of the project is to develop techniques that enable understanding of large databases of connected data. Such databases naturally form mathematical structures called graphs. Graphs are a vital way of organizing data with complex correlations. A good visualization of a graph can fundamentally change human understanding of the data. Consequently, there is a rich body of work on graph visualization.

Although many techniques are effective on small to medium-sized graphs (tens of thousands of nodes), there is a void in the research for visualizing massive graphs containing millions of nodes. Sandia is one of the few entities in the world that has the means and motivation to handle data on such a massive scale. For example, homeland security applications generate graphs from prolific media sources such as television, telephone, and the Internet.

Our purpose is to develop algorithms that enable visualization and understanding of these massive graphs. Unlike previous work, we start with the idea that in order to view the graph we must assume that individual nodes and edges are too small to be represented with the available display resolution (pixels of a computer monitor, dots-per-inch of a printout, or resolve of the human eye). For example, edges can be represented as "hairs" of minuscule width. Much like human hair, individual strands are imperceptible, but striations and highlighting make the overall structure clear.

In this way, the user can observe macrostructures in the graph and use the information gathered from a macrostructure to decide where to examine microstructures. Similar techniques were recently successfully employed to visualize streamlines, display multivariate data on parallel coordinates, and render simulated human hair.



This image shows a visualization of the large Enron graph. The concentration of the edges is represented by edge density. This image yields more information about the graph because groups of edges tend to get highlighted.

FY 2006 Accomplishments

Data Representation

Data representation is a critical part in analyzing, processing, and visualizing graphs. Our first step was to create the data representation. Our current data representation design includes a compact memory footprint (important for storing large graphs) with fast access and efficient lookups.

Density-Based Graph Visualization

A key issue with the visualization of large graphs is the tendency for the display to become quickly saturated with graph components, and a saturated display provides no information. We turned to density-based graph visualization that, instead of showing the presence of a component, is nearly guaranteed to provide visual cues to the density of the components. In large graphs with structure, common components accumulate and stand out better than the noise.

Edge Lighting
In the physical world, lighting and shading provide clues to the shape and structure of objects. This is often true of even microscopic features, for even though we may not be able to see the finer structure, it may have a profound impact on how light is reflected. These effects can be captured in an anisotropic lighting model. By applying an appropriate anisotropic lighting model to the features of a graph, we provide visual signs as to the orientation of these features.

Application Infrastructure
Although this is a research project by nature, we plan to leverage our work in applications we develop. As part of that effort, we are taking the components we develop, including much of the data structures and other scaffolding work, into a more complete information visulation ("infovis") toolkit called Titan (the Titan Infovis Toolkit for Applications and iNfrastructure). Titan is the basic toolkit for Sandia's infovis-based applications.

Significance

The development of algorithms that enable visual relationship extraction within massive graphs will enable efficient and effective information operations analysis capabilities. Consistent with our national security mission, the future domains of operation for this capability include proliferation network detection and cyber network traffic tracking.

This project will make a foundational contribution to the difficult task of extracting relationships from massive graphs. As a result, Sandia will have a unique capability to apply to difficult analytical projects for homeland security and intelligence applications. Further benefits include the establishment of Sandia as a leader in visual analytics for intelligence applications.

This project is providing key contributions to Titan for parallel, scalable information visualization. Titan is embedded in Sandia's existing framework for scalable scientific visualization, which has proven scalability and is used throughout the laboratories' day-to-day visualization needs of analysts. It will be unique in its ability to provide scalable abstract visualization and will be instrumental in current and future Sandia visualization needs.

A Dual Neutron+Gamma Source for the Fissmat Inspection for Nuclear Detection (FIND) System

93567
A. J. Antolak, D. H. Morse

Project Purpose

Active interrogation, using either high-energy neutrons or photons to stimulate fission-induced neutron and gamma-ray signals, provides an unambiguous signature of fissile material. The penetrating power of photons and neutrons allows them to "see" through intervening material that may be surrounding special nuclear material (SNM). When these particles interact with fissile material they induce fission, resulting in the subsequent emission of neutrons and gammas that may then be detected.

However, the practical use of interrogation systems has been limited due to administrative restrictions imposed on both the energy and dose of the interrogating radiation to minimize activation of or damage to the cargo. For example, photofission cross sections are generally smaller than neutron fission cross sections, so very high-intensity photon sources would be needed.

Intentional shielding used to attenuate either the source particles or the subsequently produced signals will also increase the requirements of the radiation source. Because neutrons are more efficiently shielded by hydrogeneous materials, and photons by higher atomic number materials, an active interrogation screening system should include both types of sources. Since deuterium-deuterium and deuterium-tritium neutron tube sources are in a relatively advanced state of development, we are initially exploring the development of an analog "gamma tube" photon source that uses low-energy proton-induced nuclear reactions to make high-energy gamma rays.

This project uses highly interactive modeling and fundamental experimental studies to:

- establish the scientific and engineering criteria for the dual neutron+gamma source
- determine optimal configurations and operational parameters
- validate detectability performance models using the results from scaled-accelerator experiments
- test prototypes with various shielding configurations
- determine the detection sensitivity to nuclear materials using prompt or delayed neutrons or gamma-ray signatures
- measure background levels due to prompt photoneutrons or scattered gammas from surrounding materials
- optimize the associated radiation detector system, signal processing, and analysis
- evaluate the performance and operational safety of a field-ready interrogation system (which we call the fissmat inspection for nuclear detection, or FIND, system).

The results of these experiments and simulations will define the concept of operations parameter space over which the FIND system concept is practical for detecting shielded or concealed special nuclear materials.

FY 2006 Accomplishments

Using a 700 keV Van de Graaff ion accelerator, we tested several potential (proton, gamma), i.e., (p,g), target materials and measured gamma-ray yields from these materials with a 5" x 5" NaI detector. We used a pulsed proton beam from the accelerator to induce prompt (neutron) and delayed (neutron and gamma-ray) photofission signals in uranium, which were measured with ^{3}He and NaI detectors.

Our major FY 2006 accomplishments:

- Identified lanthanum hexaboride and boron carbide as optimal target materials for ^{11}B(p,g)-based gamma tube
- Performed accelerator-based gamma-ray yield measurements and compared them to predicted values

- Tested target material robustness to proton irradiation from accelerator
- Measured prompt photoneutron and scattered gamma-ray background levels from surrounding materials
- Measured photofission-induced prompt neutrons and delayed gamma signals from depleted uranium samples
- Developed Monte Carlo n-particle transport code model of accelerator experimental setup and compared calculated results to measured photofission-induced prompt neutron count rates
- Researched, designed, and began modification of existing axial neutron tube to develop a prototype gamma tube based on ^{11}B(p,g) nuclear reaction resonance at 163 keV proton energy
- Initiated neutron yield measurements using the ^{10}B(d,n) nuclear reaction to evaluate alternative methods for producing high-energy (> 5 MeV) neutron tube sources without tritium

Significance

The objectives of this project are to perform the research and development necessary to realize a revolutionary dual-particle (neutron+gamma) source based on "nature's (nuclear) amplification," quantify its capability to detect shielded SNM, and to model the performance of a field-deployable active interrogation system that would use this dual-particle interrogation source. Recent innovations in radio frequency-plasma ion source neutron tubes will be leveraged to develop the world's first gamma tube, and subsequently both technologies will be further advanced in an integrated gamma+gneutron tube for interrogation.

The key technological breakthrough hinges on making high-energy neutrons and photons using low-energy nuclear reactions that can be reached by a simple ion source, eliminating the need for a large and complicated particle accelerator. Not only will this source be capable of operating in either continuous or pulsed modes, but it will also generate extremely intense beams of neutrons or photons for active interrogation applications. Our ultimate goal is to assess the viability of this new dual-particle source technology through fundamental experiments and simulations that determine the performance limits of the field deployed FIND system for detecting heavily shielded SNM.

Refereed Communications

D.H. Morse, A.J. Antolak, and B.L. Doyle, "Photo-fission in Uranium by Nuclear Reaction Gamma-Rays," to be published in the *Proceedings of the Conference on the Application of Accelerators in Research and Industry*.

A. Antolak, B.L. Doyle, K. Leung, D.H. Morse, and P. Provencio, "Active Interrogation Using Low-Energy Nuclear Reactions," in *Proceedings of the SPIE Annual Meeting - Penetrating Radiation Systems and Applications VII*, pp. 1-9, August 2005.

Small Acid Soluble Proteins for Rapid Spore Identification

93568
V. A. Vandernoot, T. W. Lane, A. S. Jokerst

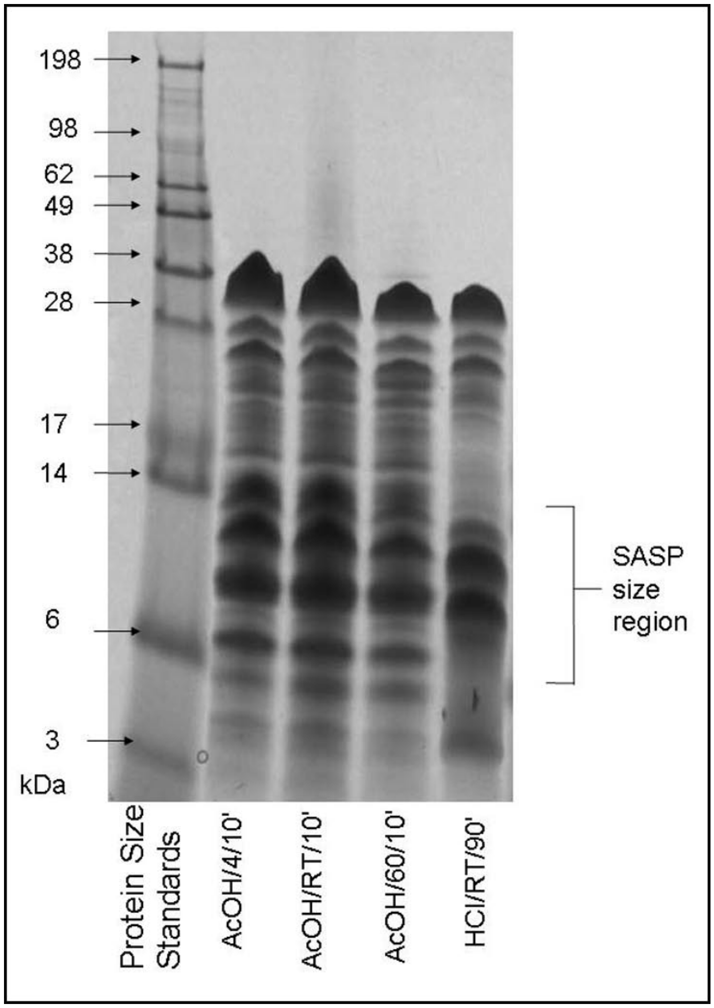
Project Purpose

This project is aimed at rapid identification of bacterial spores. Proteomic approaches are excellent complementary alternatives to nucleic acid-based techniques; they operate in scenarios problematic to polymerase chain reaction (PCR) approaches, such as samples containing polymerase inhibitors, and do not require the development and use of expensive recognition elements or primers.

A novel class of analytes that may prove advantageous for identifying spores is the small acid-soluble proteins (SASPs). SASPs are highly promising as targets as they are found in all spore formers, including *Bacillus anthracis* and *Clostridium botulinum*. They are essential for the stability of the spore (preventing genetic manipulation aimed at spoofing), SASP levels appear insensitive to growth conditions, and they are highly abundant in spores (potential for high sensitivity). Moreover, SASPs have already been successfully used to identify spores at the species level using mass spectrometry-based techniques.

This project is a one-year study in collaboration with Dr. Peter Setlow (University of Connecticut Health Center), an expert in bacterial spore research who has published numerous papers on the characterization and function of SASPs in *Bacillus* species. Our goal is to develop rapid and sensitive detection methods using SASPs to identify bacterial spores. Building on our capabilities in sensitive detection and microfluidics, we aim to devise small-scale extraction protocols amenable to microseparations and to further develop high-resolution small-protein separations.

This approach, combined with isoelectric focusing and zone electrophoresis, has great potential to allow rapid speciation of spores via their SASP patterns. Methods that separate proteins on the basis of molecular weight (chip gel electrophoresis, or CGE), isoelectric point (isoelectric focusing, or IEF), and/or charge-to-mass character (chip zone electrophoresis, or CZE), in combination with the selective extraction protocols, are expected to yield species-specific patterns of SASPs.



Comparison of SASP extraction from B. subtilis *spores using acetic acid-based protocols (AcOH) and a hydrochloric acid-based protocol (HCl).*

FY 2006 Accomplishments

Isolation of SASPs

A key aspect of our work was to develop methods for extraction that are compatible with subsequent fluorescent labeling and microseparations. The established extraction method is based on the exposure of spores to hydrochloric acid. The presence of a high concentration of acid, however, is problematic for subsequent reactions or separation steps (base catalyzed fluorescent labeling chemistries, electrokinetic injection step, pH gradient formation in IEF). As a result, we evaluated additional acid recipes based on volatile acids, including acetic and formic acid, to allow the facile replacement of the acid with a more suitable buffer after rapid evaporation.

We effectively extracted samples using acetic acid, and both the amounts and overall patterns of proteins, as evidenced by side-by-side polyacrylamide gels, were comparable to those seen with the traditional nonvolatile acid methods. This procedure was compatible with all subsequent separation and labeling steps.

We optimized the extraction methods for specificity with respect to other potentially interfering proteins. The vast majority of the proteins we extracted from the intact spore were likely SASPs (correct molecular weight range), but we saw some coextraction of the high-abundance larger proteins (in the 60+ kilodalton range). Size fractionation using ultrafiltration membranes proved successful for isolating the SASP containing fraction, in the size range below 15 kilodaltons.

Microseparations

The bulk of the separations work focused on capillary/chip-based IEF and CGE. The isoelectric points of the various SASPs covered the pH range from 4 to 10, and as a result, required broad pH range separation methods (pH 3-10). The SASPs extracted from the various SASP-producing strains of *Bacillus subtilis* were separated by IEF in capillary using commercially available IEF kits and absorbance detection.

While this approach did produce different patterns for each strain, the sensitivity of the ultraviolet absorption proved to be problematic due to the fact that the various SASP proteins did not contain any tryptophan residues, only the less-bright phenylalanine and tyrosine residues. This led to the transition to chip-based IEF in which the SASPs would be fluorescently labeled for sensitive laser-induced fluorescence detection.

The small molecular weight range of the proteins required the development of much higher resolving gels that operate in a much smaller molecular weight range. Employing modified replaceable sieving gels improved CGE resolution of small proteins.

We modified commercially available gels with varying proportions of large molecular weight polymers (typically polyethylene oxide). We found that standard small molecular weight proteins resolved better in these gels than in the commercially available recipe. SASPs of the various strains were separated by both initial conditions as well as under the enhanced conditions to demonstrate the ability to resolve. The ability to resolve the different SASP producing strains of *Bacillus subtilis* was evident as each strain produced distinct patterns by optimized CGE.

Significance

The development of rapid methods for the characterization of pathogens is of vital importance for detecting and responding to biological and chemical attacks. These techniques can have very good sensitivity; however, sensitivity comes at the expense of response time, and PCR-based techniques are problematic for samples that contain polymerase inhibitors, or are "protein only" (e.g., biotoxins). In addition, PCR-based techniques cannot detect things without developed and loaded expensive nucleic acid primers. These limitations are also characteristic of array-based or "gene array" techniques.

A proteomic approach to agent identification is a strong alternative, one that is applicable to the full threat spectrum. By targeting the proteins themselves and developing methods that evaluate the proteins directly, we eliminate the need for specific recognition or capture elements and can begin characterization, even in the case of an unknown, by searching the public domain protein databases. The majority of the research related to proteomic profiling for bacterial identification involves mass spectrometry-based approaches.

These approaches have been successful in identifying bacteria at least to the species level, in some cases to the strain level, and have the advantages of being very specific and sensitive. However, the methods can be time-consuming (e.g., tandem mass spectrometry analysis with enzymatic digestion) and, more importantly, the instrument itself is a purely laboratory device, being fundamentally too bulky to be miniaturized to a man-portable/remote site deployable instrument in the foreseeable future.

This research will allow the development of a proteomic approach to speciation that is complementary to nucleic acid-based techniques, operating even in scenarios that are problematic for nucleic acid-based assays. It promises to be rapid yet portable, making it suitable for a wide range of biodetection applications.

The use of SASPs has the potential to generate simpler but potentially more robust signatures for analysis, compared to current work aimed at whole proteome signatures. The use of microseparations for the analysis is highly advantageous in terms of speed of analysis as well as for miniaturization. This takes the analysis out of the laboratory setting and greatly extends the range of amenable biodefense applications.

The preliminary results are sufficient to form the foundation for further research and development as well as to continue to strengthen and support collaboration with Dr. Setlow. The preliminary results gained in this project should allow us to submit proposals to the National Institutes of Health or the Department of Homeland Security. Follow-on funding would enable us to design an integrated system with sample handling to allow autonomous operation and to develop a more extensive library of bacterial species, including members of the genus *Clostridium*.

Parallel Computing in Enterprise Modeling: A Hybrid Approach

93569
R. C. Armstrong, M. E. Goldsby, J. Ray, K. B. Vanderveen

Project Purpose

Enterprise modeling, social simulation, system-of-systems war gaming, and related techniques simulate complex environments and systems and compute observables that manifest themselves primarily as emergent phenomena. To increase fidelity of these systems, increasingly large computations are needed and are accessible only through high-performance computing (HPC). These simulations include models representing multiple dimensions of weapons of mass destruction detection and response, including physical entities, resources, population, and first responder decisions.

The models have a broad range of fidelity requirements depending on the phenomena of interest – they must be of sufficient fidelity to emulate significant effects yet computationally fast enough to enable timely analyses. Current capabilities, based on conventional computing architectures, are lacking in both respects. The next generation of simulation applications will require extension to HPC platforms.

We are developing a simulation code facility that will aid the creation of new parallel simulations and ease the migration of existing serial codes in this category to a parallel computing setting. The project will result in:

1. simulation components that can be mixed and matched to produce parallel or serial simulations rapidly to respond to critical needs
2. a particle-based data model that encapsulates the parallel data model, freeing other component developers from dealing with parallelism explicitly
3. a serial data model component, interchangeable with its parallel counterpart, that will still allow the simulation to run on a single processor desktop or laptop.

The resulting simulation architecture will allow the development of high-fidelity, entity-based simulation models (e.g., a moving population model for metropolitan New York City) and their incorporation into existing enterprise-level simulations. This will further enhance Sandia's ability to provide tools for homeland security and social simulation analyses, including simulation-aided development of detection, mitigation, and response countermeasures and concept of operations for terrorist attacks.

FY 2006 Accomplishments

We developed a hybrid architecture that enables existing serial Java simulation code to run on a parallel HPC platform with minimal modification. In this hybrid approach, a small number of parallel data structures and functions written in C/C++ handle compute- and data-intensive tasks, while the existing code, written in Java, remains largely unchanged. In effect, we have developed a toolkit that allows simulations written in Java and other high-level languages to interface with HPC resources.

The work thus far has concentrated on a proof of concept using code imported from a simulation of interest. We designed the parallel particle data model (PPDM) and completed the preliminary code. The Biological Defense Analysis Center (BioDAC) simulation population model has been ported to the PPDM and it is currently running on Sandia's Catalyst parallel cluster.

This simulation is running realistic entities, and the simulation code is drawn directly from the production, serial version of BioDAC. Even with population sizes that can fit on one processor, we have achieved significant speed-up (a factor of two for 8 processors). We completed a C language layer that should greatly enhance the communication performance and improve considerably the scalability for the population model simulation.

Significance

Social simulation and discrete event simulation are gaining greater recognition in management and government as essential tools that provide objective answers that no other tool can. From a scientific viewpoint, the field is in its infancy, and its effectiveness as a predictive capability will likely improve. And nothing will improve predictability more than increasing the detail and comprehensiveness of the simulation by using HPC.

Creating basic infrastructure to support parallel computations in established simulation tools will make these more comprehensive models more accessible to analysts. The infrastructure created as a part of this work will place global-scale simulations in the hands of decision makers that will be orders of magnitude more detailed and comprehensive than currently available.

Enhanced NaI Scintillation Detectors

93581
N. Bowden, W. Mengesha, M. Allen

Project Purpose

The goal of this project is to develop a high-performance radiation detector based on mature NaI(Tl) scintillator material technology. This technology could find wide application wherever NaI(Tl) is currently used; it may even allow the production of an electronics package that could be retrofit to existing NaI(Tl) infrastructure.

There has been a great deal of recent interest in developing new semiconductor and scintillation radiation detector materials. Implicit in the new material research push is that the existing materials (e.g., NaI(Tl)) have reached an asymptote in their performance, and no further improvements in detectors made from these materials are possible. However, a variety of recent technical developments open up possibilities for improving the performance of detector systems based on NaI(Tl).

FY 2006 Accomplishments

We acquired, tested, and assembled the necessary equipment to perform the experiment. We began the experiment by coupling a 16-channel multianode-photomultiplier tube (PMT) and a 16-segment NaI crystal. Using a 16-channel spectroscopic amplifier, a 16-channel constant fraction discriminator, and a 16-channel analog-to-digital converter (ADC) in conjunction with LabView software, we acquired 16 individual spectra from the NaI crystal.

Using this 16-channel system, we demonstrated the ability to align the segmented NaI crystal with the multianode PMT and record multiple spectrum from different radioactive sources (e.g., cesium-137 and sodium-22). The edge effect has a large influence on the spectra, given that only four segments – those closest to the center – are not on an edge.

After learning the fundamental procedures with the 16-channel system, we installed the necessary electronics for a 64-channel system. This required the addition of three spectroscopic amplifiers, an ADC, and software upgrades. We are aligning and calibrating the 64-channel system.

Significance

Our goal is to improve the most important parameter of all: energy resolution. We anticipate achieving this goal through a focused effort consisting of computer modeling and the application of advanced electronics and signal processing.

It has long been known that close to half of the resolution of NaI(Tl) is caused by the nonproportionality of its light yield as a function of energy. That is, electrons of different energies interacting with the crystal produce slightly different amounts of light per unit energy. Since gamma rays ultimately are detected via multiple electron interactions with a distribution of energies, this nonproportionality results in different amounts of light being observed from a monoenergetic input.

Using recently available multichannel photon detectors and readout electronics, we aim to measure the light from each electron interaction individually and, using the known relationship between light yield and energy for individual interactions, obtain the correct total energy. In contrast to other research seeking improved scintillator performance via new materials development, this approach would be relatively rapid and inexpensive.

Portable Devices for Pen-Side Disease Diagnostics

93582
W. Einfeld, J. M. Bieker, A. E. Herr, A. K. Singh

Project Purpose

No portable devices exist that enable rapid pen-side screening of farm animals for the detection of infectious diseases such as foot-and-mouth disease (FMD) in livestock. As is evident from the FMD outbreak in the United Kingdom in 2001 – which led to the slaughter of 4 million animals and an approximate $40 billion in lost revenue as a result of the outbreak – early detection and intervention during a disease outbreak can reduce economic impact and public hysteria.

Furthermore, an intentionally caused FMD outbreak is one of a number of Department of Homeland Security (DHS) scenarios receiving continued attention as a priority domestic threat. Existing methods to screen farm animals are slow, requiring two to three days in specialized labs. Furthermore, these assays cannot be used for rapid screening of large animal populations, further complicating the goal of early detection. Detection at the early disease stage is needed to minimize disease spread and economic impact.

We are developing a portable, pen-side diagnostic system for rapid screening of large populations of livestock vulnerable to FMD, including sheep, pigs, and cattle. The device uses microfluidic chip-based sample processing and antibody recognition-based identification of biomarkers indicative of FMD virus (FMDV) infection in saliva or blood. Our approach has been to target viral structural protein antigens that are indicative of an active FMD infection.

As a result of tight federal constraints associated with handling of FMDV, we are working with a number of surrogate viral species in preparation for final testing with FMDV at the DHS Plum Island facility. Further development of a robust method could eventually make possible a field-based confirmatory positive analysis, thereby eliminating time-consuming laboratory diagnostic steps prior to intervention.

We are also exploring expansion of the analytical platform to enable a diagnostic capability for avian influenza virus exposure among bird populations as well.

FY 2006 Accomplishments

The initial year of the project involved three major task areas and accomplishments as described below.

Identified a Suite of FMDV Surrogates

Since we cannot work with FMDV within Sandia facilities, we chose a set of viruses that adequately mimics the structural aspects of FMDV and presents a broad selection of commercially available antibodies for assay development. We conducted a lab safety review and ordered the suite of surrogate viruses.

Obtained Approvals to Work with BSL-2 (biosafety level 2) Viruses

The approval process proved to be a limiting factor for technical progress in the lab. We obtained Sandia approval to work with specific inactivated virus; however, we are still awaiting US Department of Agriculture (USDA) approval before we can receive shipments of inactivated surrogate virus for use in assay development. We completed all National Environment Policy Act and physical hazard survey documentation for the experimental work.

Demonstrated Proof of Concept Using a Microchip Assay System

While awaiting approval to handle surrogate virus, we evaluated microchip assay performance using a protective antigen for *Bacillus anthracis*. We demonstrated that we can separate and detect bound and unbound antigen-antibody complexes on the chip with an acceptable level of sensitivity. We also evaluated the binding characteristics of a commercially available antibody with one of our virus surrogates – *bovine enterovirus* – at a laboratory already approved for virus handling at Kansas State University, our partner institution.

Significance

Sandia is a technology developer for homeland security applications and has sponsored the development of portable devices for the detection of chemical and biological agents as well as the development of decontamination foam technologies for recovery and consequence management. Sandia also has an emerging role with the DHS in the area of agricultural security and can effectively leverage its microfluidics science and experience in this growing area.

We expect to develop this technology into a functional system with widespread application through follow-on funding from either DHS or USDA by virtue of the fact that rapid FMDV detection and response is a high priority for both of these agencies. Development of this technology also has important dual-use applications and market potential, since the surveillance of nonintentionally caused FMD in domestic herds is an important and ongoing USDA mission. Expansion of this diagnostic approach into other health science areas is also possible.

Plastic Neutron Detectors

93583

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Project Purpose

The purpose of this work is to improve the structure and electronic transport properties of organic polymers to enable particle detection applications. Specifically, sensing of individual fission neutrons via (n,p) elastic scattering in the materials may enable low-cost, large-area sensors and systems to passively detect and image special nuclear materials.

FY 2006 Accomplishments

- Achieved successful new polymer synthesis using ring opening metathesis polymerization to produce polyacetylene from cyclooctatetraene. The material is a strong dielectric, supporting fields up to 1×10^5 V/cm with no evidence of partial breakdown or excessive leakage current. The resistivity is $> 1 \times 10^{12}$ ohm-cm, enabling pulsed photoconductivity measurements with large-area samples. We demonstrated strong photoresponse characteristic of disordered semiconductors. The response is thermally activated, indicating the trap distribution is concentrated near the conduction band.
- We investigated thick polymer substrates with ordered domains.
- We improved casting and stretch orientation for poly(phenylene vinylene)-based plastic material with bulk dimensions and increased the stretching ratio from 2.5 to 5.

Significance

The new polymer exhibited properties favorable for use in electronic detectors. High resistivity and dielectric strength are extremely important milestones in the development of new materials for radiation detection.

The apparent shallow trap distribution may indicate that structural ordering in this polymer will lead to band-state conductivity similar to crystalline semiconductors. The increased elongation achieved in stretched films of commercial polymer resulted in improved charge collection.

Refereed Communications

T. Wilson, F.P. Doty, D. Chinn, A.A. Talin, M. King, L.L. Hunter, F.E. Jones, C. Cuppoletti, H. Rouchanian, and C. Muñoz, “Transport Properties of Stretch-Oriented PPV Films,” in *Proceedings of the MRS Symposium M: Conjugated Organic Materials, Synthesis Structure, Devices and Applications*, p. M3.4, April 2006.

Scintillating Nanomaterials for New Radiation Detection Devices

93584

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Project Purpose

The goal of this project is to understand how luminescent nanoparticles can be used for dynamic radiation source discrimination and to create and demonstrate a detection system based upon this understanding. Our approach is to use luminescent nanoparticles of different sizes, chemistry, and nanocomposite architecture in order to elicit a different spectral response for different excitation energies. The purpose is not to replace existing radiation detection technology but to develop new technology that increases our ability to dynamically and inexpensively monitor large areas, such as seaports and airports. This is also enabling technology for production of large surface area detectors.

All currently used scintillator materials are either bulk inorganic phosphors or polymer-based plastic scintillators. The inorganic scintillators are divided into different categories depending on the mechanism by which they emit light. Intrinsic phosphors include excitonic materials (e.g., CsI, BaF_2), self-activated materials (e.g., $Bi_4Ge_3O_{12}$, $LuTaO_4$), and semiconductors (e.g., CuI,PbI_2). Extrinsic materials include activated materials (e.g., NaI:Tl, CsI:Na, $LaCl_3$:Ce). Examples of core-valence materials include BaF_2 and CsF.

In many of these materials, there is a trade-off between critical properties such as fast decay time, luminescence output, and stability. Differential detection has been achieved using bulk $LiBaF_3$, where fast core-valence luminescence occurs under gamma ray exposure, but only self-trapped-exciton luminescence occurs under neutron or alpha-irradiation. This allows for discrimination between a gamma and neutron source.

Plastic scintillators have many advantages over conventional inorganic scintillators: they are less dense, inexpensive, less temperature-sensitive, more rugged and easily manufacturable, and have fast response times to neutrons. However, they suffer from extensive radiation damage and have significantly lower scintillation efficiencies and stopping power. Very often plastic scintillators are doped with heavy metals such as lead to enhance the stopping power. Furthermore, gamma/neutron discrimination can be accomplished via doping with boron-10. However, all determinations are made post-event.

In the quest for ideal scintillation materials, no currently used bulk material provides the simultaneous properties of stopping power, brightness, and decay time. As a result, each material is tailored to a specific aspect of the scintillation application (e.g., by tuning stopping power using a high Z elemental component). Nanocluster-based phosphors could be used to optimize the desired properties in one composite system, e.g., alloys or core/shell configurations of semiconductor materials. Stopping power could be tuned by using mixtures of high Z metal nanoclusters such as Au, Pt, or W in close proximity (i.e., nanometer) to transfer energy to the scintillating nanoclusters. This intimate mixing of immiscible materials is not possible with macroscopic, micron-size metal powders, or conventional phosphors.

FY 2006 Accomplishments

- Developed synthesis protocol for and characterization of scintillating nanoparticles/nanophosphors, including cadmium selenide, manganese doped cadmium selenide, tungsten doped cadmium selenide, cobalt doped cadmium selenide, tungsten diselenide, cadmium telluride, and tungsten.
- Developed synthesis protocol for and characterization of scintillating nanophosphor/high Z metal composites. These include: intimate mixing of tungsten nanoparticles and cadmium selenide nanophosphors and embedding of cadmium selenide nanophosphors in a silicon-tungsten sol-gel matrix.

- Designed and fabricated hardware needed for radiation testing of nanophosphor materials at the Gamma Irradiation Facility (GIF) and the Industrial Hygiene radiation protection source laboratory.
- Encapsulated nanophosphors in a sol-gel matrix and maintained photoluminescent properties.
- Optically characterized as-synthesized (liquid) and encapsulated nanophosphors under ultraviolet radiation.
- Performed initial test of spectrometer and nanophosphors using radiation excitation at the GIF.

Significance

Nanostructured scintillators should perform with near-NaI performance (both in efficiency and energy resolution) but with a less prohibitive cost. This new material would be very attractive for:

- Handheld radioisotope identifiers: Currently these devices use rather small inorganic crystals due to cost constraints. At a fixed scintillator cost, nanostructures could achieve higher efficiency, which reduces the minimum detectable quantities.
- Spectroscopic radiation portals: Most radiation portals use plastic scintillators as large quantities of detection media are needed for rapid interrogation. Using nanostructures, a radiation portal would have a much higher ability to discern between naturally occurring radioactive material, medical isotopes, and special nuclear material.
- Unattended monitoring stations: In situations where a large network of detectors is to be deployed, total system cost drives down either efficiency or the isotope resolving power of each station. A material that strikes a better compromise between these elements can raise total system performance at fixed cost.

Explosives Detection by Photoionization Ion Mobility Spectrometry

93585
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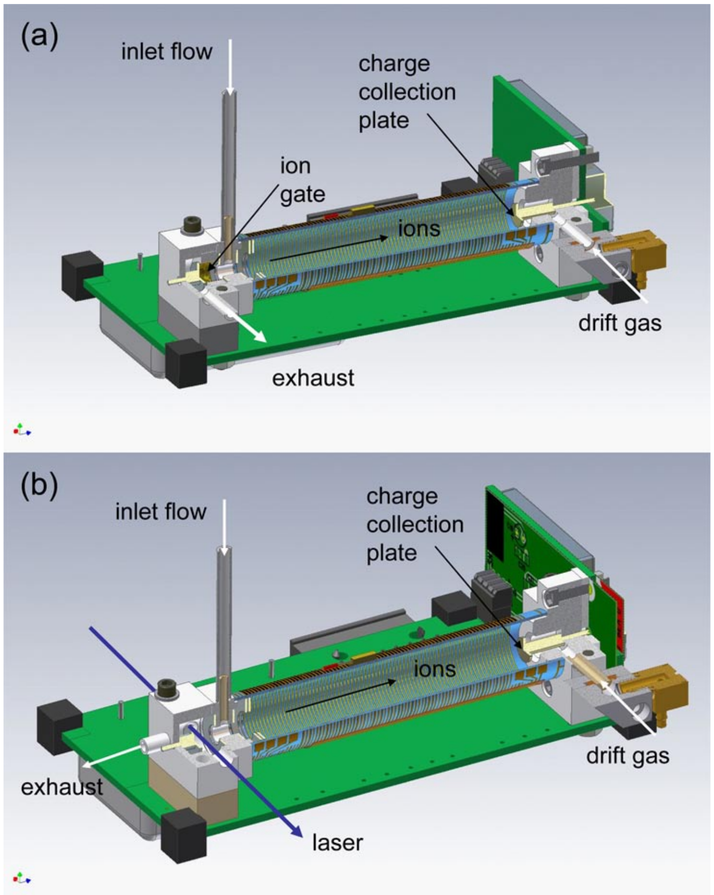
Project Purpose

Ion mobility spectrometry (IMS) allows portable, real-time detection of explosives and other chemicals. Most IMS instruments employ a radioactive ionization source, which imposes significant limitations on detection sensitivity, selectivity, and deployment. Specifically, radioactive ionization is indiscriminate, causes substantial fragmentation, and has regulatory barriers for transportation and use. Moreover, radioactive sources are continuous while IMS detectors are pulsed, which complicates the electronics, increases quiescent noise, degrades the resolution, and causes inefficient ion collection.

Resonance-enhanced multiphoton ionization (REMPI) is an alternative ionization approach that uses a pulsed ultraviolet (UV) laser to ionize the neutral analyte molecules. Lasers capable of REMPI have been too large and power consumptive to couple practically with IMS – until now. Our recent development of a compact, fiber-based, pulsed UV laser source allows us to couple IMS and REMPI and circumvent the above limitations.

We are developing a REMPI/IMS instrument for explosives detection. After initial modification of a Sandia-developed miniature IMS sensor for optical detection, we are pursuing parallel efforts to 1) optimize the REMPI detection scheme using a laboratory UV laser and the miniature IMS sensor, and 2) develop a compact UV laser to make REMPI/IMS practical outside of the laboratory. The laser and IMS sensor will then be integrated into a portable instrument based on Sandia's μHound platform.

We will also investigate the potential of extending this approach to detection of other high-priority chemicals, including toxic industrial compounds (TICs), chemical warfare (CW) compounds, and biological agents.



(a) IMS with radioactive source; (b) IMS modified for laser ionization.

FY 2006 Accomplishments

We conducted REMPI/IMS studies to determine the attainable sensitivities and specificities for detecting various high-priority explosives and for future comparison with conventional radioactive/IMS detection. We accomplished all of our FY 2006 milestones on schedule. Specifically, we:

Modified the IMS Instrument for Optical Access and REMPI (Milestone 1)

We modified a Sandia-built miniature IMS sensor for optical access (by adding ports near the inlet to the drift tube) to allow the laser beam to be focused near the IMS extraction region. Because the laser is pulsed, we removed the gate electrode on the IMS tube and the associated electronics. We performed the REMPI experiments using this sensor with a laboratory UV laser system. We modified the IMS software to acquire ion-arrival traces as single, individually triggered scans.

Demonstrated REMPI/IMS Using Standard Gas Samples (Milestone 2)
We conducted REMPI studies of nitric oxide (NO) in a bath of N_2 using an existing tunable UV laser source. We demonstrated REMPI/IMS by accessing the (0,0) band of NO, and acquired the signal (a) with the laser wavelength tuned to the spectral peak of the NO band at 226.3 nm, (b) with the laser wavelength tuned to 227.5 nm, off of the NO band, and (c) the laser tuned to the spectral peak at 226.3 nm, but with the IMS flushed with pure N_2. The presence of only a residual ion signal in (b) and (c) indicated that the signal was from NO^+.

The waveforms generated with the REMPI/IMS instrument are uncharacteristic of the typical µHound IMS waveforms. The NO^+ peak is about 5x broader than typical negative ion peaks observed in the IMS with a radioactive ionization source, and the waveform exhibits a positive-going tail that is not expected from positive ions. This waveform shape does not change with either laser power or NO concentration. The peak width is similarly broad just after the initial capacitive transimpedance amplifier, indicating that the broadening is not caused by the subsequent signal-conditioning electronics.

We constructed a second µHound IMS that included the ion focusing components removed in modifying the first IMS; however, we did not observe narrowing of the resulting peaks. We are now investigating the possibility that the waveform widths are due to diffusion in a section of the drift tube in which ions experience no electric potential.

Demonstrated a Q-switched Fiber Laser Using a Bulk Pockels Cell (Milestone 3)
The compact tunable UV laser system for the portable REMPI/IMS instrument is based on a rare-earth-doped fiber laser. We constructed and evaluated two different Q-switched fiber lasers: 1) with a linear cavity and a Faraday mirror to obtain a linearly polarized output beam, and 2) with a ring cavity with two half-wave plates to establish linear polarization.

Analyzed the Nonlinear Frequency-Conversion Scheme (Milestone 4)
We designed a frequency-conversion scheme to convert the near-infrared fiber-laser output to the UV wavelengths necessary for REMPI.

Significance
IMS is finding increased use for detection of explosives, CW agents, and illegal drugs, with an estimated 70,000 IMS units in service worldwide. Combining IMS with REMPI will support a variety of laboratory missions by enabling sensitive and selective, and therefore advanced and improved, portable detection of explosives and related compounds. The system will have applications in weapons (propellants, explosives), nonproliferation, military technology, and homeland security. REMPI/IMS will therefore be of significant interest and utility to existing and potential customers such as the Department of Defense, the intelligence community, law enforcement, and the Department of Homeland Security.

IMS is also frequently used for process analytics. Although IMS is not generally used to identify unknown compounds, it is increasingly being used to monitor processes in which the expected compounds are known. IMS is especially attractive for field and process applications because ambient air can be used as the carrier gas. Such applications include monitoring of gas insulated substations, water quality, and the odoration of natural gas, as well as diagnosing human breath composition and metabolites of bacteria.

Given the broad scope of IMS applications beyond explosives detection, REMPI/IMS could be extended to detect other high-priority substances, including CW compounds, TICs, and biological agents.

Detection of Cell Phone and Wireless Systems

93586
R. A. Salazar, L. D. Bacon

Project Purpose

Cell phones pose a security threat to Sandia information because a person using a cell phone can transfer classified information out of the technical areas undetected.

The purpose of this project is to provide added protection of Sandia information. To achieve this goal, we are developing technology that detects cell phones. The immediate application of this technology is to prevent the introduction of cell phones into the technical areas. The extended application is the detection of other banned wireless systems into Sandia's technical areas. This technology can provide an added layer of security at any site where cell phone use introduces a security threat.

To accomplish this goal, we are developing technology that uses radio frequency (RF) radiation to interrogate a person entering the technical area in order to detect forbidden electronic devices. If an electronic device receives the RF radiation, it will re-emit RF radiation. All electronic devices use nonlinear components. The nonlinear components – located within the electronics – that absorb RF energy reradiate it in an altered state.

The reradiated RF energy contains information that identifies the electronic device. The major identifiable characteristic of the reradiated RF is that it contains energy in the original transmitted frequency and in the harmonic frequencies. Detection of harmonic energy indicates the presence of a nonlinear component (and hence an electronic device) and in some cases can identify the class of electronic device.

FY 2006 Accomplishments

We tested cell phones, pagers, and two-way radios, as well as noncommunication devices such as digital integrated circuits (ICs) and simple circuits composed of a single nonlinear component. Results of these tests show that an instrument using radiated RF can detect wireless systems. Noncommunication systems also reradiate RF but have a fundamentally different response than communication systems, enabling discrimination between the two classes of systems.

Each test consisted of radiating continuous wave RF radiation onto a device-under-test (DUT) while recording reradiated emissions. Each DUT re-emitted harmonics; the range of the harmonic content included the fundamental up to the ninth harmonic. The pattern of re-emitted radiation was different for each DUT, making it possible to distinguish one DUT from the others.

We conducted each test in an anechoic chamber to enhance the signal-to-noise ratio, thereby making detection of each DUT easier. An instrument under normal operation would not have the luxury of improved signal-to-noise provided by an anechoic chamber, but it would have the benefit of improved signal-to-noise ratio provided by advanced signal processing and knowledge of devices intended for detection and rejection.

Significance

This technology will enable Sandia security forces to introduce a layer of protection not currently available and integrate it into turnstiles or entrances to monitor for possession of unwanted wireless devices. This technology could provide Sandia with a distinguishing capability of interest to industry and government.

Virtual Security Design, Analysis, and Training Tool

103389

G. D. Wyss, P. G. Kaplan, N. P. Orlando-Gay, P. B. Merkle, J. L. Mitchiner, M. K. Bhardwaj

Project Purpose

The objective of this "feasibility" project is to identify the technical hurdles and gaps involved in collaborative security analysis activities and understand whether tool creation and adaptation can address those gaps. We conducted an interdisciplinary security demonstration exercise with relevant after-action analysis.

FY 2006 Accomplishments

We identified both technical and nontechnical issues that thwart effective collaborative security analysis. We referred the nontechnical challenges (e.g., logistical and classification issues) to management for study and resolution. The technical challenges we identified relate to ontological/semantic issues, geospatial analysis issues, and data access/data structure issues for the large and diverse databases that must be traversed quickly for tactical security challenges. We documented these issues and their potential resolution methods.

Objectives we identified for future work include developing and instantiating a grammar or semantic ontology to enable interrelationships of concepts, data, and tools among diverse risk analysis domains, as well as developing qualitative and/or quantitative methods to estimate and/or rank the likelihood of attacks for particular targets.

Significance

We identified technical hurdles and gaps for collaborative security analysis activities and laid the groundwork for the next phase of collaborative-integrated security analysis research and development activities. We expect that collaborative security analysis techniques will enable us to answer currently intractable security risk analysis problems in the areas of homeland security, infrastructure security, nuclear materials, and weapons security, among others, by enabling the full spectrum of risk causes, mitigators, and effects to be integrated in a risk-informed decision analysis methodology.

Emergency Preparedness for Biological Attacks

103390

L. I. Yang, D. D. Djordjevich, N. Spencer, A. S. Yoshimura

Project Purpose

Sandia developed BioDAC (biological decision analysis center), a simulation and decision-analysis tool, to help model the detection of and response to a biological attack on a major US city. Among other things, BioDAC models the operations of different responding organizations (including hospitals; federal, state, and local governments; first responders; and the military) and the effect these operations have on the unfolding attack and its aftermath.

BioDAC could be greatly improved with more detailed and realistic modeling of hospital and other medical operations, particularly in the areas of emergency medicine and prophylaxis distribution. Professor Aaron Bair of the University of California-Davis Medical Center and Dr. Nathaniel Hupert of the Weill Medical College, Cornell University, have been researching and modeling hospital emergency room (ER) operations with the goal of producing detailed, accurate predictions of how a given-size ER would respond to a given input of patients, including triage times and throughput.

Additionally, they have developed detailed simulations of prophylaxis-dispensing operations in response to biological attacks or epidemics, which are also parameterizable. Both Prof. Bair and Dr. Hupert have won widespread recognition within the medical and emergency preparedness communities for their work. Inclusion of their models (or at least the content thereof) would greatly bolster the accuracy of BioDAC's modeling, as well as the credibility of the BioDAC within the emergency preparedness community.

Building on Prof. Bair's and Dr. Hupert's models and inserting this important capability into BioDAC will not be easy. BioDAC is implemented in Java, while Prof. Bair's model is implemented in the Extend modeling language, and Dr. Hupert's model is implemented as an Excel spreadsheet. Another challenge is that the level of granularity of the models is somewhat different. Prof. Bair's ER model simulates at the level of a single hospital, and likewise Dr. Hupert's model concerns the operation of a single prophylaxis distribution center. BioDAC, on the other hand, models all of the hospitals in a metropolitan area, as well as the entire prophylaxis distribution response, but does not model internal hospital or distribution center operations to the degree of fidelity as the other two models.

Because of the differences in implementation language and degree of fidelity, BioDAC executes much more quickly than the other two models for a given period of simulation time. Maintaining BioDAC's speed of execution while integrating the other two models' content will be yet another challenge.

To overcome these challenges, we will work with Prof. Bair and Dr. Hupert to develop an approach to understand and integrate the content of their respective models into BioDAC, if not the actual models. This may take the form of developing reduced fidelity models based on their models. The reduced fidelity models would most likely be implemented in Java and then integrated into BioDAC.

FY 2006 Accomplishments

We enhanced PopulationDAC and the broader set of DAC tools by incorporating content from external higher-fidelity models of health care operations and disease. We also added a pandemic influenza outbreak model to PopulationDAC in anticipation of future systems analysis needs for this relevant and timely concern. We tested these external models and conducted limited analyses and comparisons between the external models and enhanced DAC models.

This project provided, in addition to DAC enhancements, an approach for future model development that draws from external models. Incorporation of higher-fidelity, enhanced models into DAC enables users to conduct more detailed analyses that lead to improved understanding of decision making and recommendations for emergency preparedness and response.

Significance

Sandia's DAC simulation tools are used in systems analyses and exercises to provide guidance on emergency preparedness and response. Population-DAC is one version of DAC that models health care response to public health emergencies. It models an urban area, including its geography, population, and health care resources and operations. It then overlays a threat scenario – e.g., a disease, caused by a bioterrorist attack or outbreak – on the area, and calculates consequences to the population based on decisions made over the course of the scenario. This tool allows the user to analyze emergency health care response options and the impacts of decisions on scenario outcomes. Outcomes include metrics, such as numbers of fatalities and numbers of people provided with health care.

Tunnel Detection

103392

N. P. Symons, D. C. Craft, H. D. Nguyen

Project Purpose

Tunnels under the US-Mexico border have long been a major route for illegal drug smuggling. The Drug Enforcement Administration (DEA) and Border Patrol in California and Arizona have identified large tunnels with sophisticated infrastructure. A major tunnel recently discovered in Otay Mesa near San Diego, CA, is approximately half a mile long and has sophisticated infrastructure and construction. A Sandia team participated in a data collection "rodeo" at this tunnel, where both seismic and long period electromagnetic (EM) data was collected.

The purpose of this project is to analyze the existing data collected from the Otay Mesa tunnel and determine if useful features can be identified for developing tunnel detection methods. We obtained passive seismic data collected from three sites, one within the border easement over the deepest part of the tunnel, another in a seismically quieter site midway between the border and the tunnel entrance (construction site), and data from immediately outside the building containing the tunnel entrance during periods of tunnel activity. We also obtained the active source data collected at the first two sites using a 100# accelerated weight drop (AWD) source, and EM data recorded at the second site.

FY 2006 Accomplishments

We performed analysis and signal processing on the passive seismic data recorded at the building site.

After examining the data from the easement and the construction site, we determined that the recording time was too short for testing of the passive Green's function method. Based on this analysis, we developed new software (within Matlab) to acquire data for very long time periods and to generate a windowed cross correlation "on the fly" instead of recording the data. The advantage of this new method is that we are now able to produce cross-correlation data sets at much higher sample rates than have been attempted in other studies. We are acquiring continuous data (for approximately six weeks) at a Sandia site, as the ground in the Albuquerque area dries out, in order to demonstrate the utility of the passive Green's function method to measure changes in the subsurface material properties that might be indicative of an illicit tunnel.

Significance

Several federal agencies (US Army, Center for Security Evaluation, State Department) have contacted Sandia over the last several years requesting our expertise in the search for tunnels under the US-Mexico border and around US embassies in foreign countries. Although we have a great deal of experience in developing techniques for the location and characterization of larger-scale facilities in denied areas, we have had no experience in and have limited data regarding this new environment. This effort will put us in a position to develop the technology necessary for the solution of this vital homeland security/defense issue.

Heterogeneous Distributed Network Sensing Feasibility Study for Security Applications

103393

H. D. Nguyen, J. R. Van Houten, D. I. Toledo, J. W. Giron

Project Purpose

The purpose of this project was to demonstrate an integrated capability in a cost-effective multilevel architecture that combines Sandia-developed technologies with low-cost, mass-produced, commercial off-the-shelf (COTS) sensors. The key elements we addressed using COTS products were communications, data processing, and both feature-based and decision-level fusion. These elements contribute to the overall multilevel approach and support the heterogeneous sensing and distributed processing that will enhance detection, classification, and tracking of asymmetric (human, nonmilitary vehicles) threats in complex terrain.

Specifically, our project focused on algorithms enabled by the network sensors to detect and track humans moving covertly (slowly, as to mask presence in background noise) through the sensor field. We focused only on the seismic signatures of a person moving through a sensor field as datum points for analysis, knowing that other phenomenology, such as imagers, is available.

Our interest and focus was to use COTS sensors with a little computing power to process as much as possible and to combine and share data at a much higher level. The higher level in this case was a platform with higher computational horsepower and fidelity sensing, such as array processing and image processing algorithms. These sensing algorithms could be triggered by low-level nodes sending a decision or a feature set to be processed by the high-level node.

With the emergence of cost-effective commercial systems, networks can be created that contain a mixture of elements ranging from dumb sensors to extremely smart sensing nodes. Driven by the commercial availability of low-cost and low-power sensing nodes (e.g., motes by Intel), sensor fields on the order of 100s to 1000s are now possible. However, past research by the Defense Advanced Research Projects Agency has focused on an operational paradigm that consists mainly of many homogenous nodes that are similar in function (i.e., they lack computational resources to perform more complex operations). These low-cost nodes have been used mainly in monitoring sensing schemes, with very little processing done at the node level.

Attempts to use a variation of these homogeneous sensor nodes to process and refine data gathered met with mixed results. Our premise is that heterogeneous sensors that combine more capable nodes with the low-cost motes nodes in distributed sensing applications will enhance detection and tracking in broad-area situational awareness in complex terrain for security applications. Our project focused on improving existing Sandia sensing technologies using distributed algorithms enabled by the network to enhance detection of personnel and reject false alarms caused by the environment and animals.

FY 2006 Accomplishments

Algorithm Performance

We integrated a fixed-point algorithm to detect footstep signatures and to extract features for classification at a higher-level processing node.

Ad Hoc Communications

We established an ad hoc mesh network using XBOW motes and demonstrated autonomous infrastructure establishment under real-world-like environmental conditions that include deployment, range, and spatial coverage areas.

Power Measurements

We derived and validated a power model to predict life span of the low-cost nodes in relation to different algorithm parameter settings such as sample rate, duty cycle processing, averaging window, and transmission power in the communications system.

Test Demonstration
We demonstrated functionality in walk tests and established a detection range and preliminary false alarm hypothesis.

Significance

The results of this project will impact current and future approaches to network sensing for security applications. These breakthroughs will provide additional situational awareness to fixed facilities to both enhance force effectiveness and become a force multiplier in a sense as optimal resource allocation can be achieved through sensing and fast response. Commercial sensors, in conjunction with Sandia algorithms and architecture, will provide a robustness and reliability not possible with homogenous solutions. The significance will be that we can cover more ground for less cost using industry technologies that are similar in nature.

Military Technology and Applications

Weaponization of Thermobaric Explosives

67032

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Project Purpose

The purpose of this project is to develop a general-purpose enterprise-scale logistics support system modeling capability. This capability is unique in that it models the complete enterprise of a military platform: the supply chain, the repair chain, usage of the platform, on-platform maintenance, tracking of all parts and their repair status, and a global deployment. It will also be used to determine inventory levels for the full run of the simulation, uptime statistics on the military platforms, status of all parts, costs by site, and shipping times.

We introduced some randomness into the model to reflect part failures, shipping time variations, improper repairs, missing parts and resources, and so on. The model reproduces the same results each time it is run given the same random seed. Additional features augment the model to allow optimization of parameters. The optimization may be external to the program itself, such as wrappers around the simulation that optimize particular parameters of interest. Nonetheless, the model makes the data available for these optimization routines and uses updated parameters in subsequent runs.

Finally, the model is generic and flexible enough that it can be tailored to the simulation of a wide variety of military platforms as well as run data from specific platforms as tests of the code. Such a test case was the Joint Strike Fighter (JSF), which has been tested on another enterprise model called SEM (support enterprise model). This provided a means for validation of the current model.

The user interfaces make it convenient to enter data as well as analyze output data. As an agent-based model, it has the further advantage of being scaled into a parallel model, resulting in large time savings per simulation run versus run times on a single processor.

FY 2006 Accomplishments

We completed several tasks toward the goal of an agent-based military logistics enterprise model:

We finished the basic architecture design for the agent-based model. The model is now capable of simulating a large range of military logistic enterprise applications. It can model supply and repair chains, tasks at each site (such as repair), the allocation and usage of support equipment (resources and personnel) for these tasks, on-platform maintenance, usage of the platform (which models the aging of parts), and the tracking of the complete status of every part and every platform throughout the simulation. The agent-based architecture leverages the work of the NISAC (National Infrastructure Simulation and Analysis Center) team on the economics infrastructure interdependency model N-ABLE (NISAC Agent-Based Laboratory for Economics).

We used a small-scale test problem consisting of a basic military aircraft supply and repair chain for validation. We ran the model with data from an example used by SEM. Though much debugging was needed, we succeeded in duplicating generally accepted results for this test case, providing much needed validation of our model.

We modified the software to run a large-scale test problem, in this case, the enterprise modeling of the JSF. Though the model remains flexible enough to be applied to other applications, we invested a significant amount of work in taking the very large database of the JSF logistics enterprise and converting it to run on our agent-based model. This involved developing parsers and pruning techniques to enable the database to feed into the model without overwhelming the memory capabilities of the computer. Results were mixed. Though our model showed the supply and repair chain for each part, most of the aircraft exhibited more downtime than with SEM. The discrepancy is most likely due to the need to prune the input file to reduce its size to be able to initialize. This means that one or more parts most likely have their supply/repair chains disconnected from the squadrons needing the parts. This problem remained unresolved at the time of the project completion.

We completed the parallelization effort. The model runs on the institutional cluster (liberty) for an arbitrary number of nodes. We did not observe a speedup in performance for the small test cases, primarily due to the lack of any coincidence in time of tasks for these small test cases, but we believe we will see significant speedup with the larger test cases (including JSF).

We formalized the optimization strategy. The objective function is a combination of inventory and transportation costs along with maintenance of a certain level of weapons system availability. The general strategy is to use an analytic model as an approximation of the full agent-based model, and then to use a mixed integer linear programming approach on the analytic model for the global optimization.

Significance

We completed several key tasks leading to the development of a realistic weapons system enterprise prototype. Our general agent-based weapons logistics model is not tied to any particular application and is capable of simulating a range of applications with only input file modifications. Thus, it should be useful to a range of military applications such as the JSF, Future Combat Systems, Navy ships, and so on.

In addition, because of its flexibility, it could be considered for applications outside the military, such as the Global Nuclear Energy Partnership (GNEP), health care networks, commodity flow networks, and other socioeconomic enterprises. Further, with its ability to work with heuristic optimization packages it could be used as a decision support system in all these applications. Finally, the parallelization capabilities of the model will enable faster run times for simulation, which is especially useful in optimization scenarios.

Our parallelization investigations revealed much useful information regarding the potential for speedup. The nature of parallel models is to distribute processing of the model to multiple processors. This works best when the agents do not process too much information sequentially, i.e., one does not gain much if the agents have to wait on each other before moving on to the next task. Further, the communication overhead of agents messaging to one another is a significant constraint on performance. Therefore, the speedup from parallelization of an agent-based model is highly problem dependent.

If the communication overhead is not too steep, and many tasks can be distributed rather than occur in lock-step progression, then the speed up from parallelization is significant. But, as was the case with the small test file, communication overhead is large compared to processing time. Thus, parallelization was not beneficial. We hypothesize that the large JSF test case will show better performance, but we have not performed this test.

Another alternative to parallelization on a cluster (which we shall call distributed memory architectures, or DMA) was to investigate shared memory architectures (SMA). Although rare in the marketplace, Sandia has expertise in multithreaded SMAs. As of this writing, several Sandia researchers in this area believe that SMAs may be a better avenue for parallelization of agent-based models (including military logistics) than DMAs. If this area shows promise, it would put Sandia at the leading edge in parallelization of agent-based models. It would also provide the kind of speedup for enterprise applications we have not currently been able to show using the institutional cluster. This will be investigated further.

Refereed Communications

P. Krishnamurthy, F. Khorrami, and D.A. Schoenwald, “Computationally Tractable Inventory Control for Large-Scale Bidirectional Supply Chains,” in *Proceedings of the 2006 American Control Conference*, June 2006.

Novel Processing, Affordable Motion Compensation, and Mode Multiplexing for Miniaturized Synthetic Aperture Radar

67035

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Project Purpose

There is a need for high-performance radar with a size and cost compatible with small UAVs (unmanned aerial vehicles) and next-generation precision munitions. Consequently, Sandia has ongoing efforts to develop miniaturized, affordable radar hardware (miniSAR and MESASAR programs). The goal of this project has been to investigate equally innovative control, motion measurement, and image formation architectures that would fully exploit the new hardware's capability. From a big picture perspective, this project has had a significant role in evolving high-performance radar from an exotic sensor on limited platforms to a ubiquitous tool available to the ever-growing fleets of small UAVs.

The focus for this year has been twofold: 1) create and validate FPGA (field programmable gate array)-based image formation algorithms, and 2) investigate and validate motion measurement enhancements based on autofocus algorithm improvements as well as novel IMU (inertial measurement unit) calibration schemes (i.e., rotational indexing).

The purpose of the first task is to facilitate real-time video synthetic aperture radar (SAR) and fine resolution strip map modes in next-generation systems. Meeting this goal requires an increase in effective processing density of about 60 X (compared to the general purpose processor used in miniSAR). Specifically, we worked to demonstrate that radar algorithms can be migrated from processor-based code to an FPGA-based implementation, and that tremendous gains in effective processing density are achievable.

The purpose of the second task is to facilitate the use of a smaller, lower-cost IMU in next-generation radar systems without degrading the resulting image quality or resolution (IMU cost and size scale with performance). Specifically, we aimed to demonstrate that an order of magnitude reduction in motion measurement error for microelectromechanical systems (MEMS)-based IMUs can be obtained through simple indexing schemes.

Real-time SAR image of golf course (4 inch resolution at 4 km range) from Sandia's miniSAR radar, using novel, LDRD-developed software architecture.

FY 2006 Accomplishments

The FPGA-based image engine design consists of two major design blocks: the image formation block and the autofocus block. Each block in turn consists of multiple subblocks. FY 2006 accomplishments include the following:

- We designed all subblocks and completed their realization as Virtex IV FPGA intellectual property. Several subblocks include novel FPGA-based approaches that vary significantly from previous software-based approaches.
- We modeled and simulated all subblocks and then validated them in commercial evaluation boards. This overall design represents the most complex FPGA IP simulation ever attempted at Sandia.
- We performed full end-to-end validations for the entire design in a piecemeal fashion. Results indicated that the full design will achieve the desired 60 X increase in processing density.

- We completed a high-level layout for the custom board.

The IMU indexing evaluation study involved both empirical measurements and extensive simulations. FY 2006 accomplishments include the following:

- We performed laboratory tests to determine the benefits of indexing a MEMS-based IMU. The results clearly indicated that an order of magnitude improvement is achievable in both gyro and accelerometer performance.
- We used a six degree of freedom trajectory and motion measurement simulation code to investigate the effect of indexing on inertial navigation errors for a representative aircraft flight environment. (These simulations give an indication of how the motion measurement errors would affect airborne imaging radar.) The indexing technique shows significant promise in reducing both heading and attitude errors. The preliminary conclusion is that indexing appears to allow an instrumentation-grade IMU to function almost as well as a tactical-grade IMU.

We continued to investigate improvements to the enhanced autofocus algorithm. Specifically, we investigated techniques to reduce the required processing time. For example, one technique that shows significant promise involves using image domain convolutions with a focus correction kernel. We implemented the algorithm in Matlab code and successfully tested it against actual radar phase histories. Further investigation is necessary to fully realize this innovation's potential.

We continued to investigate additional improvements to the radar system/control architecture (developed in FY 2004/05). Two examples include the following:

- We performed a study to evaluate the optimal flight path an aircraft might take in making a stripmap image of a road with moderate curves and direction changes.
- We investigated what radar control modifications might be needed for operation on a small UAV.

Significance

For real-time operation, Sandia's miniSAR currently only offers a spotlight mode. (No current system, large or small, offers a real-time video SAR or fine resolution stripmap mode). The tremendous significance of the image engine effort is that it will facilitate real-time high data-rate modes (such as video SAR and fine resolution stripmap SAR) without increasing the radar's size or weight. These modes would greatly enhance a radar's utility and facilitate entirely new applications. Such a capability is highly desired by the user community.

Moreover, the technical achievement demonstrated by successfully migrating the very complex image formation and autofocus algorithms to the FPGA domain is in itself a highly noteworthy achievement. In achieving this goal, we developed a robust process that will greatly benefit future efforts.

High-performance imaging radar requires high-performance motion measurement. A tactical-grade IMU (LN200) is currently one of the most expensive components in miniSAR. The significance of the IMU indexing investigation is that it could ultimately facilitate a 3 to 5 X reduction in IMU cost and weight by allowing an instrumentation-grade IMU to achieve tactical-grade performance. The highly positive results from this year's simulations and tests indicate that this goal is likely achievable.

Similarly, the enhanced autofocus algorithm allows the final processing to compensate for more severe motion measurement errors than previously thought possible. This in turn directly lowers the performance requirements of the IMU. Other efforts will continue this investigation, with the end goal of actually using a less expensive, smaller IMU in current and next-generation radar systems.

The system architecture developed in this project is also demonstrating significant value. This architecture both facilitated the operation of miniSAR and provided it with unprecedented flexibility. Many customers have expressed an interest in miniSAR, and several new, major projects are now in progress. The modularity and robustness of the novel system architecture can now be exploited to meet the needs of a diverse customer set.

Other Communications

D.F. Dubbert, A. Sweet, G.R. Sloan, and A. Doerry, "Results of the Sub-Thirty-Pound, High-Resolution MiniSAR Demonstration," in *Proceedings of the SPIE Defense and Security Symposium*, p. C2090, April 2006.

G.R. Sloan, "MiniSAR Update," presented (invited) at NMIA/AIE UAV Payloads Conference, Washington, DC, May 2006.

G.R. Sloan, "MiniSAR for Small UAV Reconnaissance," presented (invited) at NMIA/AIE UAV Payloads Conference, San Diego, CA, December 2005.

Enhanced Perception for Remote 3D Mapping of Unknown Indoor and Outdoor Environments

67037

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Project Purpose

The simultaneous localization and mapping (SLAM) generic problem generally refers to a robot in an unknown environment that is attempting to generate a map, localize within the map, and estimate the uncertainty in sensory information. The goal of this project was to develop two-dimensional (2D) and three-dimensional (3D) simultaneous localization and mapping algorithms. The 2D algorithms are based on laser range image data, while the goal of the 3D effort was to use inertial measurement data combined with video to perform the localization.

FY 2006 Accomplishments

We successfully demonstrated 2D simultaneous localization and mapping on a mobile robot that employed a scanning laser range finder. In addition, we developed 3D localization algorithms and demonstrated them with a camera-inertial measurement unit connected to a personal computer.

Significance

3D simultaneous localization and mapping can be applied to mobile robot navigation, security and surveillance missions, and accident response missions. The 3D capability developed under this project can be used for indoor navigation and mapping without the aide of a global positioning system (GPS), which is normally available only outdoors. In urban areas, GPS is often not available, so SLAM is the only way to navigate in an unknown environment.

MICROFUZE Integration

67039
M. L. Chavez, A. S. Tappan

Project Purpose

The primary mission of a weapon fuze is to provide system safety enablement and subsequent control of weapon detonation events. Within this architecture, a fuze must integrate inputs from various environmental sensors and, if the preset conditions for weapon function are met, supply the initiation stimulus for detonation. Evolving mission scenarios for future DOE and Department of Defense (DoD) weapon platforms, such as for hard and deeply buried target defeat (HDBT) and "smart bullets," will soon outpace the capabilities of today's fuze technologies.

Targeting buried assets and meeting tomorrow's battle-field engagements demands advanced fuzing architectures and technologies characterized by "intelligent" function under multiple, high-g impacts. Achieving increased fuze functionality in a size constraint implicit with high-g survivability implies novel system architectures, incorporation of new concepts in munitions safety, and the realization of miniaturized integrated components based on emerging processes and technologies.

In a previous LDRD project, novel fuzing technologies necessary for enabling a microelectro-mechanical system (MEMS)-based miniature fuze were established. In that project, ground-breaking technical progress was achieved with demonstration of a multilayer deep reactive ion etched mechanical safe and arm device that allows out-of-plane motion and responds to two separate high-g mechanical environments, low-voltage microinitiation technologies, and the innovative processing and detonation characterization of an explosive train on the MEMS size scale.

This project seeks to integrate the previously developed microinitiator and explosive train technologies with the MEMS mechanical safe and arm device to enable a true MEMS fuze. The project will culminate in the successful firing and detonation of an artillery shell, under field test environments, resulting in the first fully-integrated MEMS device to ever accomplish such a feat.

FY 2006 Accomplishments

We continued to place emphasis on military standard safety compliance and system-level integration of the key MICROFUZE technologies in order to allow field-level technology demonstrations. However, our development activities unfortunately encountered a set back when the multilayer metal process that we were using became unavailable. The company (Microfabrica Inc.) discontinued its involvement in our efforts. We chose an Albuquerque company, HT Micro Inc., to continue the project. HT Micro's processes made it possible to deliver multiple MICROFUZE designs with a relatively short lead time.

Specific FY 2006 accomplishments:

1. We investigated the feasibility of using beam bending theory to accommodate for the different capabilities the new microsystems manufacturer could achieve. This method showed promise theoretically by allowing for refinement of the systems geometry, allowing us to optimize our system to meet the safety compliance standard.
2. We designed, fabricated, and tested the first build of the safe and arms devices. Receiving these devices allowed verification that the manufacturer could produce the devices needed for the planned experiments. Duplicate MICROFUZE devices were built using two different material compositions: pure nickel and nickel iron alloy. Testing was conducted using a centrifuge system at Sandia that was capable of obtaining data via framegrabbing software that outputs real-time video footage. Results showed that the nickel iron alloy is a superior material for our application, having a yield strength of at least nine times that of pure nickel.

3. We designed, fabricated, and tested multiple devices with self-contained shuttle assemblies. This MICROFUZE iteration was designed to allow for multiple testing of each device and to enable determination of the reliability of the data. The data again showed the great potential of the nickel iron alloy which did not yield under the stresses the Sandia centrifuge continuously created.
4. We created a self-containing shuttle that integrated a thin gold membrane required for explosive deposition.
5. We advanced the noncontact, fluid-based deposition process for integration of the primary explosive into MICROFUZE to the point where consistent deposition of primary explosives on arrays of multiple substrates is well-developed. We consistently deposited the primary explosives, lead azide and silver azide, into substrate arrays as large as 3 x 5, and there is no reason the process cannot scale to much larger arrays.

We scaled up the synthesis of silver azide to the 500 mg quantity and verified the fine-particle characteristics of this material. We performed characterization experiments on sub-mm columns of these explosives and observed limiting diameter effects.

We confirmed direct detonation transfer from the deposited primary explosive to a lead pellet, representing the next explosive increment. We conducted this initiation with a slight gap between the reactive bridge ignitor and the primary explosive, representing the necessary gap for slider travel in a real fuze. While we conducted only a preliminary characterization, we proved the necessary framework for a viable, manufacturable microexplosive train.

Significance

With the continued development of deposition technologies and fundamental characterization of microexplosives, Sandia continues to take a leading role in the area of microenergetics. The deposition technology under development in this project has led to much interest in the energetic materials community and enhances Sandia's capabilities for future research and development. Furthermore, the MICROFUZE safe and arm development efforts continue to emphasize fuze design and safety criteria set by DoD. By developing safety hardware based on MEMS thin film technology, Sandia looks to remain the leader in field of micromechanics.

Other Communications

A.S. Tappan, P.C. Jung, E.J. Welle, and R.J. Pahl, "MEMS-Compatible Processing of Energetic Materials," presented at the 50th Annual Fuze Conference, Norfolk, VA, May 2006.

Analysis of Technology Impacts on Operations in Complex Environments

67041

M. J. McDonald, B. E. Hart, D. H. Hart, K. J. Page, B. P. Van Leeuwen, S. Tucker, E. P. Parker, F. J. Oppel III

Project Purpose

The purpose of this project was to develop a unique capability to assess technology impacts in urban (and urban-like) peacekeeping, warfighting, and security environments. When this project was initiated, there were no effective analysis tools to support the evaluation of technology impacts on operations in complex physical environments such as facility security, homeland security, and military operations in urban terrain (MOUT). The key issues are the tight coupling between complex urban environments and the technologies, along with the variable fidelity modeling capabilities needed to support analysis breadth and depth.

In one scoping of needs in 2002-2003, a Defense Advanced Research Projects Agency (DARPA) Senior Advisory Group (SAG) performed a wide-ranging search for analytical tools and models addressing MOUT. They received little response and judged what existed as fragmented and insufficient. Their consensus was that fixing the problem was hard. A similar message was heard within the physical security and homeland security communities. Recognizing a needs overlap that we were uniquely positioned to address, we developed a process to evaluate TTPs (tactics, techniques, and procedures), CONOPS (concepts of operation), and strategies against new threats, new technologies, and new and old environments and facilities.

Based on these findings, we defined and organized this project around a technical goal of dramatically improving simulation accuracy, breadth, and fidelity for analyzing technology impacts in these environments. To provide a lasting science and technology legacy, work was also planned to address Sandia's larger need for a sophisticated analytic capability for complex systems engineering. Here, complex system models are enablers for complex systems engineering in that analysis is the foundation of science-based engineering. As analysis must consider the operational environment, this project included building a modeling and simulation environment to support such engineering.

By its close, this project met its goal and made significant and credible progress. We developed new technologies of integration, identified several emerging Sandia codes for inclusion, and extended and integrated these codes as well as key commercial codes into the resulting environment. The Sandia codes include models for secure ad-hoc communication, sensor network phenomena, unmanned air and ground vehicles, and human elements. Commercial codes include Department of Defense-preferred communications codes (OPNET), terrain editing tools (Terrex), human motion simulators (DI-Guy), and high-level architecture (HLA) federation codes. Existing urban and DOE facility terrain models are being used for analyses.

FY 2006 Accomplishments

We developed and tested technologies necessary to simulate and analyze complex adaptive systems in complex environments. Our accomplishments include expanding the Umbra model library; describing new communications algorithms for energy-constrained ad-hoc wireless networks; supporting the concept of having modeling and simulation algorithms, data, interactions and structure match operational system of systems elements; and scientifically demonstrating that the Umbra framework enables efficient, cost effective, and time effective implementation of large-scale, multidisciplinary, multiattribute, complex simulations.

We also demonstrated conclusively that simulation is a necessary partner to experimentation, with the potential to generate significant savings in time, effort, and expense when designing, examining, and testing the performance of complex adaptive systems.

In FY 2006 we completed:

- Final Summer Study
  - Purpose: Push state of art in integration
  - Built upon convoy scenario from generative modeling
    - Investigated the addition of ground sensing and tracking to locate attackers
    - Investigated the use of remote-operated guns on vehicles
    - Used a robotic vehicle range scenario to complete the first test case for asset integration, demonstrating that our representation facilitates integration of diverse sets of models
    - Used Results in organic aerial vehicle and precision attack missile experiments for modeling and simulation commonality
  - Used results in technology integration for final MOUT analytics demonstration

- Model and Federation Design Enhancements
  - Developed OPNET HLA federated object model
  - Used descriptor fields to pass data between objects – this made it easier to develop scenarios and pass information.
  - Development simultaneous in Umbra and OPNET
  - Put infrastructure in place for the OPNET federation design, the basis for new federation services, with addition of new features built in Umbra.

- Model Improvements
  - Communications (new verified groupcast algorithm)
  - Facility analysis sensor models (long- and short-range radar)
  - Basic human representation (variable-fidelity)
  - Simplified behavior engine development
  - Tracking (Talon algorithms enhanced)
  - Detailed analysis of new communications algorithms
  - Provided experimental basis for low-powered networking
  - Filed technical advance (TA) on new communications algorithms

- Modeling Wrap-Up and Final Reports
  - SAND report and TA on efficient communication protocols
  - Algorithms for groupcast concept/scaling
  - Low-power sleeping modem algorithms
  - New and refined networking algorithms to address scaling
  - New analytic approaches for modeling communications

Significance

The key accomplishment of this project was a significant modeling and simulation capability for addressing a broad range of complex systems design, analysis, and engineering tasks. In particular, we developed a general capability to model, simulate, and analyze advanced military and security technologies, their CONOPS, and the TTPs for using them in complex urban and security environments. Additionally, we developed new representations that made it easier to model these and other complex systems of systems that operate in heterogeneous environments. Finally, our analysis and experiments show that the product we produced supports a new degree of scalability: scalability over domains or degree of heterogeneity.

This development is significant in terms of the types of systems that Sandia can now analyze in a tractable form. For example, security sensor systems operate in many sensing domains or modalities, such as physical contact, electro-optics or infrared, magnetic, acoustic, and seismic properties. Networked sensors operate in radio frequency and communications domains. The targets they sense operate and interact with each other and their environment in other domains. We demonstrated that for some modeling solutions, model complexity grows geometrically with the degree of heterogeneity.

In situations Sandia is interested in, this complexity limits the analytic tractability. Problems become too complex to model. Conversely, the Umbra framework supports abstractions that allow the model complexity to grow nearly linearly with heterogeneity. Problems that were once intractable can be built on relatively small budgets. We recognized, built tools to exploit, leveraged the opportunity, and demonstrated this effect. A general result is that these methods can and are being exported to domains beyond this work.

A second accomplishment was in the development and refinement of the models themselves. We brought together a significant number of models and optimized them for complex systems analysis and then reused them in their original and in successor tasks for significant gain. In some cases, we used the models to discover new engineering concepts. A particularly notable product is a set of three new, novel, and publishable communications algorithms, which are described in a SAND report and in a TA.

Finally, we demonstrated that simulation can provide a deep understanding of both algorithms and experimental design. In many cases, such as the communications protocols, it is not humanly possible to track the number of details that would need to be studied for an adequate "by hand" analysis. In the case of complex environments such as urban military operations, fielding experiments, collecting data, and correlating the results is a cost-prohibitive activity when compared to the simulation alternative. We concluded that simulation is a necessary partner to experimentation, with the potential to generate significant savings in time, effort, and expense when designing, examining, and testing the performance of complex adaptive systems.

Tools we developed are already being used for applications at nuclear weapon facilities. Applications include a remote weapon system simulator, advanced sensor system engineering tools, an augmented reality-based special weapons response team training system, and a battle simulation tool for the design and analysis of neutralization technologies.

Other Communications

E. Parker and J.B. Rigdon, "Structural Similitude in Simulation Effectiveness: Enabling Systems-of-Systems Engineering from Concept through Production," presented at the NNSA Future Technology Conference, Washington, DC, October 2006.

M.J. McDonald, B. Hart, and F.J. Oppel, III, "Description and Analysis of New Low-Power Ad-Hoc Networking Algorithms," Sandia Report, SAND 2006-6066, Albuquerque, NM, August 2006.

Systems Analysis of Networked Sensors

67047

D. A. Wiegandt, A. R. Church, B. McDaniel, D. E. Gallegos, F. K. Wunderlin, P. E. Sholander, M. T. Oswald, K. J. Smart, D. Kilman, E. Ollila, D. K. Steele

Project Purpose

The main impact of this project is in the development, analysis, and simulation of robust, adaptable, and energy efficient wireless sensor network architectures. Our FY 2006 goals were to complete the analysis and simulation of adaptive, large-scale, heterogeneous sensor networks. We defined the wireless sensor network architecture and simulation space in previous years. We completed significant work on analyzing and simulating wireless sensor networks for systems design. We enhanced the project goals to include actual implementation of project theory to prove worth in the embedded system. We also implemented and proved low-power wake-up and routing.

We applied an analysis process for wireless sensor networks to a large-scale data exfiltration application. The system analysis focused on using multiple communications links based on local sensor sub-network clusters to increase system throughput. The process included steps of first-order analysis, topology modeling, and network simulation of the distributed algorithms and communications. We implemented adaptive methods within an embedded system in order to prove larger data file passing techniques that we had analyzed earlier in the project.

The systems analysis process produced novel results. We completed application-specific analysis to define the problem within the network architecture and simulation space and to provide baseline performance estimates. Next, topology analysis of clustering algorithms narrowed the design space. This analysis ruled out regions of the parameter space that will not produce working systems. We summarized and documented the findings of the graph theoretic analysis, topology analysis, and network simulations.

FY 2006 Accomplishments

We developed a sleep-mode protocol for an energy-efficient wireless sensor network for event detection, target classification, and target tracking. It was used in conjunction with the dynamic source routing algorithm in order to reduce network maintenance overhead, while providing a self-configuring and self-healing network architecture. We developed a method for determining the optimal sleep time.

We analyzed a sleep-mode protocol that employed a cyclic radio sleep protocol at the node level and protocol parameters that could be optimized for a given application. Cyclically placing a network radio to sleep (in a low-power state) reduces power consumption and increases latency. In an effort to mitigate latency, protocols such as STEM (sparse topology and energy management) employed a pair of simple radios, one for data and the other for communications. We looked at protocols that used a single network radio. While the use of multiple radios did show power savings and reductions in latency, several problems existed with that approach.

Using the sleep protocol as a theoretical guide and the research that was combined to create the protocol, we developed a practical implementation of it within an embedded system. Methods developed in year one and two for efficient large file transfer were also implemented within the embedded system to enable efficient routing and use of large files.

Significance

The work accomplished through this project paves the way for efficient implementation within embedded real-world sensor networks. The low-power wake-up schemes enable future continuation work to develop the necessary methods for minimizing power consumption. Specifically, power consumption should be centered around processing of sensor information and less on routing of the information to the collection point. By minimizing the power consumed in link setup and information passing, these systems can effectively use their battery capacity for information processing.

Simulations of the routing and data exchange, in addition to the low-power wake-up methods, were key to implementing a sound design in the embedded system. The hardware proof of this project provides a significant foundation for further development of this process.

Refereed Communications

P. Sholander, M. Oswald, B. McDaniel, D. Gallegos, E. Ollila, D. Wiegandt, and D. Steele, “Systems Analysis of Networked Sensors: Final Report,” Sandia Report, SAND 2006-6695, Albuquerque, NM, October 2006.

Deployable Object Tracker for NMD Flights

67048

T. A. Pitts, D. L. Davidson, M. W. Koch, J. V. Sandusky, R. O. Nellums, L. A. Jones, W. J. Zubrzycki, A. D. Niese

Project Purpose

The principal purpose of this project was to determine the viability of developing a system to characterize the relative kinematic and dynamic properties of a deployed object from a remote platform via laser radar measurements. Such a system would not impact the flight physics of any of the target objects but would provide data currently available only through on-board instrumentation. Specifically, we proposed application of the scannerless range imager (SRI) technology to problems related to national missile defense (NMD) concerns in space-based deployments.

Electronics and instrumentation on board NMD test targets affect flight kinematics and dynamics. If it is possible to acquire the necessary measurements from a remote location or platform, then more realistic (and potentially less expensive) testing is possible. Remote measurement via three-dimensional (3D) ladar does not require the cooperation of the target and can operate at extended ranges. Also, acquisition of 3D surface information allows classification of unknown objects. These measurements require the ability to acquire measurements on multiple objects in the sensor's field-of-view.

The key to determining target flight physics is the ability to acquire sufficiently accurate and rapid samples of the target's location and pose relative to the sensor. We proposed acquiring and analyzing data on moving targets with the SRI ladar. This mature technology provides high-speed, high-resolution 3D imaging.

Sandia has significant experience with scannerless 3D ladar. Sandia sensors are currently operating at the Laser Radar Range Facility at Eglin Air Force Base and the Nonacoustic Underwater Imaging Branch of the Naval Coastal Systems Station. They have flown on the space shuttle for the structures directorate of the National Aeronautics and Space Administration (NASA) Johnson Space Center and more recently as sensors for the NASA Shuttle Return-to-Flight missions. While all of these sensors are capable of high-speed, high-resolution imagery, pose estimation techniques critical to determination of an object's kinematics and dynamics had not been previously applied to data of this type.

FY 2006 Accomplishments

This fiscal year, we:

- Constructed a motion stage and control system, including software control and automated data collection capability. The system hardware and software provide full six-degree-of-freedom motion capability, and extensive data collection has exercised four degrees of freedom. The stages are sufficiently accurate to provide truth data on the programmed trajectory for comparison with computationally estimated pose. A specific trajectory may be programmed with automatic data collection occurring at specific locations or continuously along the specified path.

- Collected several pose data sets (targets with both specular and diffuse surface properties were used) with an SRI engineering development unit, using the aforementioned automated collection system. Models are sufficiently small to fit entirely in the sensor field of view.

- Collected and analyzed rotation and translation data on two full-scale satellite models (with both specular and diffuse surface properties). The data was collected at a Naval Research Laboratory facility using the on-site motion control system.

- Investigated several pose estimation algorithms. These were principally variants of Hough transform and ICP (iterative closest point) methods. We determined that ICP methods were the best candidates for real-time in situ understanding of object kinematics.

- Developed pose-estimation software based on ICP algorithms. The software is capable of loading, displaying, and aligning large 3D data sets, while reporting a complete set of target pose parameters for later display and analysis.

- Investigated both specular and diffuse target surfaces. Diffuse target surfaces provide smoothly varying Lambertian returns and are excellent candidates for pose estimation via laser radar modalities. Specular targets are more challenging as they require higher dynamic range systems and/or methods of reducing the dynamic range of the received data. We investigated transmit and receive channel cross-polarization glint abatement techniques as possible solutions to this challenge.

Significance

The data collection and analysis have demonstrated the viability of remote pose and location determination for in-flight or on-orbit applications. Systems capable of such measurements have numerous applications in NMD and space-based deployment areas. Interest shown by potential government and nongovernment customers indicates a clear recognition of the need for this capability.

Experimentation and data analysis have identified what we believe are the principal measurement and signal processing challenges for application of laser radar to real-time pose estimation for determination of target kinematics and dynamics. These are specular surface properties and multipath. Development of methodologies and modalities capable of dealing with these difficulties would likely provide sufficient advancement to construct a robust fieldable system.

The data collection and analysis capabilities developed in this project are uniquely suited to future development of this technology for specific applications and investigating solutions to various technical challenges.

Simulating Human Behavior for National Security

71943
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Project Purpose

This project focused on a significant technical gap in existing modeling and simulation capabilities: the representation of plausible human cognition and behaviors within a simulated environment. Specifically, the intent of this project was to demonstrate initial virtual human interaction (HI) modeling capability that realistically represents intra- and intergroup interactions between virtual humans and human-controlled avatars as they interact with their environment.

To accomplish this, we made significant process in simulating human behavior in a manner that produces realistic characteristics and movement. We developed the simulations from scientifically rigorous research-derived models based on current experimental research in cognition, perception, physiology, and cognitive modeling. We enhanced our existing cognitive models with additional research to include culturally plausible behaviors most important for representing human interactions. We implemented these models in a modular, interoperable, and commercially supported simulation framework (Umbra).

Instead of using production rules that drive behaviors of simulated humans, the HI framework uses levels of activation of perceptions, concepts, and states. Production rules are efficient and appropriate for architectures that are chiefly concerned with behavioral control and predictability. That is, behaviors derived from production architectures are guided by "if-then" rules, which state that if an objective is to do something, and the parameters have been satisfied, then it will perform an operation associated with that objective. The HI framework seeks predictability as well, but it puts a much greater emphasis on modeling the cognitive processes that ultimately guide the behaviors of simulated humans.

It is asserted that rigorous modeling of cognitive processes will ultimately provide more accurate, predictive behaviors. For example, the HI framework allows for multiple perceptions, goal states, and action intentions which can concurrently have some degree of activation. Once a perception has been activated by cues in the environment, it may trigger a specific, intermediate goal state if that state is consistent with higher-level goals and current environmental conditions. The intermediate goal that is activated will trigger an action intention state. The action intentions that are ultimately chosen will be mediated by current emotion states (i.e., fear, anger) that are affected by what they perceive in their environment.

FY 2006 Accomplishments

This fiscal year we increased the psychological and physical realism of our avatars and the complexity of the simulation environment. We demonstrated this accomplishment in an intelligence gathering scenario that featured three interacting simulated humans whose high-level "cognitive" behaviors were driven by cognitive models. The setting was a market square within a city in Iraq. In the scenario the market square had been used to store and transfer illegal arms to enemy forces. US solders received a tip that a market vendor had a weapons cache and were instructed to search all vendor stands.

The behaviors of the US soldiers are controlled by a user via voice commands. The user can order, by name, one, several, or all of the troops to investigate a particular person. Two of the vendors, as well as a vendor's wife, have cognitive models that enable them to dynamically respond to the actions and communication of the soldiers. The number of US soldiers present, the communication style they employ with the vendors (i.e., dismissive vs. respectful of their culture), and the gender of the soldiers affects the behaviors of the cognitive avatars. For example, a male soldier talking directly to the vendor's wife could anger both the wife and the vendor. Conversely, a female soldier talking directly to the vender may anger both the wife and the vendor.

The simulated humans can sense each other, react to each other, and move about in the simulated environment. The cognitive-model-driven simulated humans perceive patterns of cues from the environment and respond appropriately to different environmental situations. These simulated humans also have dynamic emotion states that are perturbed by what they see in their environment. That is, if their environment changes so do their perceptions regarding that environment, and consequently their emotions and behaviors change as well. Furthermore, the simulated humans have preliminary action generation or motor-level cognition models that transform abstract actions generated by high-level cognition to actions for execution by a simulated physical human model.

We extended the Umbra sensing simulation subframework to include cognitive-level properties and sensing/perception of relationships among entities. We developed and implemented a preliminary subframework for simulating the emotion state of a cognitive model and a preliminary modeling capability for the effect of the emotion state on context recognition. We also enhanced the capabilities of the cognitive framework by introducing a new context pattern type that is useful for comprehending situations in which multiple instances of a property or relationship are perceived.

On a software engineering level, the entire project code base was modularized into a standard form that enables other groups to start developing and using cognitive-model-driven simulated humans in other Umbra-based projects.

Significance

This work integrated, and thus leveraged, past investments that included Umbra and the Cognitive Modeling Framework, resulting in a modeling and simulation capability that will serve as the technical foundation for subsequent national security analysis and security personnel training. It is also anticipated that this technical foundation will ultimately provide security enhancements and/or new technologies that will aid in formulating better security strategies. Moreover, the capabilities generated by this project will serve as the technical foundation for next-generation training tools for DOE response forces and DoD military personnel.

Using human simulations for training is an effective way to reduce costs. Unfortunately, current training simulations do not have the resolution and fidelity to replicate the asymmetries often seen in the real world. Simulated human behaviors in current training simulations tend to be brittle, and students can easily "game the system" after learning its limitations. A potential benefit of using a training simulation with the HI framework is greater psychological realism. The simulated humans could have the cognitive model of foreign adversaries, including the mindset, behaviors, and training typical to a particular country or geographical area. This is especially important because complex combat scenarios are shaped by political, military, and economic developments.

The use of HI models could eventually provide our forces with additional tools for dealing with an evolving threat through action/counter-action forward simulations. We could create simulated humans that interact with each other and their environment in a realistic manner that simulates friendly/adversarial behaviors. For example, there could be a situation that threatens the security of the facility (such as an angry protest crowd). The response force could use the tactics development tool to help predict behaviors several generations before they occur. This would provide an environment to evolve new threat understandings and to provide red/blue-force reasoning engine.

Moreover, the HI models could ultimately forecast the movements and general behaviors of large groups of individuals in response to US soldier actions, and so on. This would be useful in helping predict population responses to the transportation and/or protecting of sensitive materiel. In fact, HI models are currently being used to help simulate the cumulative economic effect of people as a result of natural disasters.

Advances in computational cognitive modeling could be integrated into intelligent systems. In particular, the cognitive framework can be viewed as a hybrid symbolic- and neural network-based system. There is also substantial potential for cross-fertilization between the cognitive-model-driven simulated humans and cognitive robotics.

Micro-Optical Radar (MOR) Facial Recognition Project

73207

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Project Purpose

The purpose of this LDRD is to improve security at US points of entry through the development of next-generation three-dimensional (3D) biometric identification. The fundamental enabling technology will be micro-optical radar (MOR) technology coupled with advanced recognition algorithms. The objective of this project in FY 2006 was to test the Katana 8 sensor ASIC (application specific integrated circuit). This ASIC included an array of 4x4 pixels. Evaluating the Katana 8 ASIC will provide data and technical insight for future ASIC designs that incorporate a much greater number of pixel processors.

FY 2006 Accomplishments

We tested all electrical functions of the ASIC and found all 16 channels to be operational. The interpixel modulation phase was stable and repeatable. Virtually no cross talk was observed between adjacent pixels. We also explored alternatives to improve the manufacturability of the ASIC for volume applications.

Significance

Successful operation of the Katana 8 ASIC is a major step towards the realization of 3D flash focal plane array sensors for applications such as 3D facial recognition. This technology has widespread application in both government and consumer markets. Although one of the most complex high-performance mixed mode ASIC designs ever produced, the current design demonstrated function and feasibility.

We developed new layout techniques to reduce noise, as well as alternative flash ASIC designs for future exploration. We formulated several new concepts that may be explored in future efforts such as LDRD, work for others for other federal agencies, or cooperative research and development agreements.

Adaptive, Peircean-Based Decision Aid

79829
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Project Purpose

This project aimed to integrate a number of technologies into a framework that supports decision making from a basis grounded in mathematics and logic theory as opposed to the "band-aid" approaches that exist today. Integrating formal concept analysis as the knowledge representation technology permits reasoning theoretics to be applied against the knowledge bases constructed. The logic applied is based on a Peircean model that includes abduction as a key component of the hypothesis generating mechanism.

This theoretic construct was integrated with a co-evolutionary game engine to provide a new predictive capability. Problems faced by analysts do not have patterns that can be used as situational "flags" in an information landscape. Modern intelligence problems no longer are amenable to classic inductive-deductive technologies. The integration of these technologies and the engineered solutions provide a verifiable environment to anticipate novel adversarial actions.

FY 2006 Accomplishments

We developed the core analytic infrastructure that supports Peircean-based decision aids and began developing the coevolutionary game engine. The formal concept analysis implementation extends the ideas of Ganter-Willie by explicitly defining the interaction operator in their theory with a set definition based on "predicts."

This exploration permits an analyst's decision aid to be built in a way that permits reasoning to be performed on blocks of knowledge structured along taxonomic lines. The structure and implementation of the core algorithms also support the reasoning operators defined by a number of researchers in the field. The abductive system can learn as well as reason from a causal perspective, supporting a number of unique reasoning functions that typically reside in disparate computational systems. Additionally, we defined a graphical front end that makes the job of representing information to an analyst less complex.

Significance

We demonstrated that it is possible to define a decision aid that is mathematically robust and uses a knowledge representation mechanism that supports transformation into alternative mathematical domains that are optimal for a broader spectrum of knowledge manipulation technologies.

We moved the state of the art of knowledge representation, manipulation, and formalism beyond the "fault-tree" methodologies of current information approaches that simply fix weaknesses when they are discovered. Our approach provides a logical and mathematical basis for making design decisions that mitigates many weaknesses prior to implementation into a system solution.

Refereed Communications

M.E. Senglaub, "Fusion Subsystem Design," presented at the ICCRTS, San Diego, CA, June 2006.

M.E. Senglaub, "Fusion Subsystem Design from an Integrated Command, Decision Support, and ISR Perspective," presented at the ICCRTS Conference, San Diego, CA, June 2006.

Other Communications

M.E. Senglaub, "Formal Concept Analysis," presented at the Department of Homeland Security Signatures Program Review, Arlington, VA, September 2006.

Design Tools for Complex Dynamic Security Systems

79831

K. N. Groom, B. R. Rohrer, J. J. Harrington, D. G. Wilson, J. B. Rigdon, R. H. Byrne

Project Purpose

The goal of this project is to develop tools that enable the design of complex dynamic security systems. The three characteristics of a dynamic security system are: detection beyond the usual boundaries, adaptation to changing threats, and a reduction in the predictability of the response. Complexity in autonomous agents is often identified by the appearance of emergent or complex behavior from relatively simple rules. We will investigate complex theory concepts that may be incorporated and designed into existing dynamic security systems to enhance the stability and performance.

We plan to investigate several novel technical approaches to complexity over the course of the project: partially observable Markov decision process (POMDP), exergy/entropy nonlinear control design (a methodology for analyzing complex systems) and percolation/graph theory. In addition, we plan to develop a dynamic security system test bed at Sandia's Robotic Vehicle Range (RVR) to validate the algorithms and tools developed under this effort. Although we will focus on the dynamic security application, our goal is that these tools will generalize to other classes of complex systems.

FY 2006 Accomplishments

100-Node Wireless Sensor Network (WSN)

- We deployed and tested the WSN at the RVR, which provided the primary component of our dynamic security test bed. We organized the units into a 10 x 10 grid, with 15-yard spacing. Each node consisted of a radio, seismic sensor, battery, and solar cell, as well as firmware for operation and communication.
- We equipped each node with an external pyroelectric infrared (PIR) sensor head. Each head consisted of four PIR sensors facing in the cardinal directions, each with a 90 degree field of view.
- We implemented a custom annunciator program to display sensor trips and control the WSN. The program uses the Umbra simulation environment for three-dimensional visualization.

POMDP

- We applied POMDP to the problem of sensor scheduling in wireless sensor networks. Sensor scheduling is the coordinated control of sensors to minimize power consumption and target tracking error. POMDP allows for a nonmyopic control policy by considering future opportunities in the decision process. We used particle filtering for state estimation and Q-value approximation to solve the POMDP.
- We extended POMDP to the case of tracking multiple targets in a wireless sensor network. We employed joint probability data association (JPDA) to match sensor hits with the correct intruder.
- We extended POMDP to allow multiple sensors to track a target. We fused data using multisensor JPDA (MS-JPDA).
- We ported POMDP and MS-JPDA to the Umbra simulation environment.

Exergy/Entropy

- We developed a novel nonlinear control system design methodology based on exergy/entropy thermodynamics, applicable for Hamiltonian systems, and stability characterized with Lyapunov direct methods and optimal analysis. The result is both necessary and sufficient conditions for stability and performance of nonlinear systems.
- We used the exergy/entropy design methodology to analyze and test SISO (single input, single output and MIMO (multiple input, multiple output) nonlinear systems. Using the same methodology, we investigated its application to large groups or collectives of dynamic systems.

These results were for both regulator and tracking control systems.

- We established exergy sustainability designs that provide a missing link in the analysis of self-organizing systems: a tie between irreversible thermodynamics and Hamiltonian systems. As a result of this work, the concept of "on the edge of chaos" is formulated as a set of necessary and sufficient conditions for stability and performance of sustainable systems.

Percolation/Graph Theory

- We investigated graph theoretic techniques for measuring the robustness of various networksand showed that algebraic connectivity is not as useful as previously thought for measuring graph robustness. In particular, algebraic connectivity can increase greatly, while node and edge-connectivity actually decrease. The results were accepted for publication in *IEEE Control Systems Magazine.*

Significance

100-Node WSN Test Bed

Sandia does a great deal of work in nuclear weapons security and in supporting Department of Defense-based security. WSNs provide security forces the ability to monitor large areas and base perimeters around the clock with minimal manpower. While flat areas can easily be monitored with cameras (infrared and visible wavelengths), rough terrain, woods, swamps, and so on cannot. Inexpensive sensor nodes can be densely placed in these areas, thus, providing tracks of intruders entering the controlled area.

Beyond its clear application to security, the 100-node WSN can be used to study the interactions of large numbers of autonomous agents. Understanding such complex interactions is important to many diverse fields, from critical infrastructure to atomic interactions.

POMDP

We applied POMDP to the problem of sensor scheduling in large WSNs in order to optimize global objectives such as overall power consumption and intruder tracking error. Such issues are critical to a fielded WSN system. The need to track multiple targets through a network is also an important real-world problem that we solved using MS-JPDA techniques.

Porting these techniques to the Umbra environment allows high-fidelity simulation of security scenarios, which is useful for assessing installations. POMDP uses Monte Carlo simulations to determine the best decisions. Therefore, a higher fidelity simulation will improve its optimization.

Exergy/Entropy

The exergy/entropy methodology provides a unique set of criteria for designing nonlinear controllers for nonlinear systems with respect to both performance and stability. Traditionally, almost all modern control design is based on forcing the nonlinear systems to perform and behave like linear systems, thus limiting its maximum potential. Our novel nonlinear control design methodology overcomes this limitation.

The exergy/entropy methodology could revolutionize how control systems are designed. The field of control plays a critical role in many areas, such as manufacturing, electronics, communications, transportation, computers, and networks, as well as many commercial and military systems. This methodology is applicable to the design and control of complex, multicomponent and often adaptive systems of arbitrary purpose, design, and underlying physics, such as critical infrastructure (electric power grids, telecommunication and satellite systems, oil and gas pipelines) and military systems (network centric warfare, critical assets/nuclear security systems). Secure and reliable operation for all these systems is vital to a healthy economy for both civil and military infrastructure.

Refereed Communications

R.D. Robinett III and D.G. Wilson, "Exergy and Irreversible Entropy Production Thermodynamic Concepts for Control System Design," in *Proceedings of the IEEE International Conference on Control Applications*, October 2006, CD-ROM.

A. Clauset, H.G. Tanner, C.T. Abdallah, and R.H. Byrne, “Controlling Across Complex Networks,” to be published in the *IEEE Control Systems Magazine*.

Y. Li, L.W. Krakow, E.K.P. Chong, and K.N. Groom, “Approximate Stochastic Dynamic Programming for Sensor Scheduling to Track Multiple Targets,” in *Proceedings of the 5th Workshop on Defense Applications of Signal Processing*, December 2006, CD-ROM.

R.D. Robinett III and D.G. Wilson, “Exergy and Entropy Thermodynamic Concepts for Nonlinear Control Design,” in *Proceedings of the ASME International Mechanical Engineering Congress and Exposition*, November 2006, CD-ROM.

Y. Li, L.W. Krakow, E.K.P. Chong, and K.N. Groom, “Dynamic Sensor Management for Multisensor Multitarget Tracking,” in *Proceedings of the 40th Annual Conference on Information Sciences and Systems*, pp. 1397-1402, March 2006.

R.D. Robinett III, D.G. Wilson, and A.W. Reed, “Exergy Sustainability for Complex Systems,” *InterJournal of Complex Systems*, no. 1616, August 2006.

L.W. Krakow, Y. Li, E.K.P. Chong, K.N. Groom, J. Harrington, and B. Rigdon, “Control of Perimeter Surveillance Wireless Sensor Networks via Partially Observable Markov Decision Process,” in *Proceedings of the 2006 IEEE International Carnahan Conference on Security Technology*, October 2006.

R.D. Robinett III and D.G. Wilson, “Exergy and Irreversible Entropy Production Thermodynamic Concepts for Control System Design,” in *Proceedings of the IEEE International Conference on Robotics and Automation*, May 2006.

R.D. Robinett III and D.G. Wilson, “Exergy and Irreversible Entropy Production Thermodynamic Concepts for Control Design,” in *Proceedings of the Nonlinear Regulator Systems, 8th IASTED International Conference on Control and Applications*, May 2006, CD-ROM.

R.D. Robinett III, D.G. Wilson, and A.W. Reed, “Exergy Sustainability for Complex Systems,” in *Proceedings of the International Conference on Complex Systems*, June 2006, CD-ROM.

R.D. Robinett III and D.G. Wilson, “Exergy and Irreversible Entropy Production Thermodynamic Concepts for Control System Design,” in *Proceedings of the Nonlinear Systems, 14th Mediterranean Conference on Control and Automation*, June 2006.

R.D. Robinett III and D.G. Wilson, “Exergy and Entropy Thermodynamic Concepts for Control System Design,” in *Proceedings of the AIAA Guidance, Navigation, and Control Conference*, August 2006.

Other Communications

A. Clauset, H.G. Tanner, C.T. Abdallah, and R.H. Byrne, “Controlling Across Complex Networks,” Sandia Report SAND2006-2661J, Albuquerque, NM, April 2006.

R.D. Robinett III, D.G. Wilson, and A.W. Reed, “Exergy Sustainability,” Sandia Report SAND2006-2759, Albuquerque, NM, May 2006.

Knowledge Discovery via Sensor Fusion in Structures and Ad-Hoc Networks

79832

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Project Purpose

Situation awareness (SA) continues to be an important technological challenge in homeland security and defense scenarios. For applications such as force protection, an effective decision maker needs to maintain an unambiguous grasp of the environment. The purpose of this project is to explore and create computational mechanisms for the adaptive fusion of diverse information sources.

Our research involves the use of neural networks and Markov decision processes to provide SA assessments given a set of information sources. Furthermore, the system operator's input is used as a point of reference for the machine learning algorithms.

The amount and diversity of data from which to make SA assessments, and the ways in which to interact with human decision makers in need of SA, are difficult problems that we address in our research. We pursued technological capabilities for decision support with the decision maker as a central and balanced element of the system. We blend three focus areas: machine learning, information fusion, and decision support.

The machine learning components provide adaptive mechanisms for information fusion, situation awareness, and the ability to continuously sense and influence the state of the environment. Information fusion offers the ability to integrate diverse data sources into meaningful and actionable information. Finally, decision support addresses the need to facilitate objective, effective, and consistent human performance.

Our distinctive computational SA approach uses different machine learning mechanisms (unsupervised, supervised, and reinforcement) in one system. The capability to switch learning methods based solely on change in data type, with no human intervention, will bring machine learning to a new level of robustness and task-adaptability. Research and development of optimal information fusion/SA in the presence of hostile adversaries is an important aspect of our work.

FY 2006 Accomplishments

In collaboration with the University of Missouri Rolla, we accomplished the following:

Sensor Fusion/Situation Awareness Design

Our approach uses different machine learning mechanisms in one system. The design of our computational engine for sensor fusion and situation awareness integrates elements of unsupervised, supervised, and reinforcement learning. The capability to switch learning methods based solely on change in data type, with no human intervention, will bring machine learning to a new level of robustness and task-adaptability. The design matches extremely well with the needs of situation awareness and human-in-the-loop decision support.

Sensor Fusion Experiments

We used a rich set of Defense Advanced Research Projects Agency SensIT Program data (obtained from a repository held at the University of Wisconsin), to develop and test sensor fusion methods on realistic force protection problems. We conducted experiments to determine the direction of a vehicle using decision fusion from the detections of 23 sensors distributed along three roads. These experiments initiated our work to develop methods that will be robust in the midst of malicious adversaries. We are also developing decision fusion algorithms to determine vehicle speed, location, and heading.

Cyber SA

Our investigations into the notion of situation awareness in cyberspace resulted in a situation awareness definition paper and a cyber SA concept outline.

Significance

The project resulted in technology/research that is

- relevant to Sandia, the military, and the nation
- complementary with other Sandia activities, primarily virtual perimeter security, Protective Forces projects, Defense Threat Reduction Agency projects, and Umbra activities
- innovative, pushing the envelope of machine learning technology by combining multiple learning methods into one architecture.

A distinctive aspect of our approach is the use of different machine learning mechanisms in one system. The design of our computational engine for sensor fusion and situation awareness integrates elements of unsupervised, supervised, and reinforcement learning, and matches extremely well with the needs of situation awareness and human-in-the-loop decision support for the following reasons:

- Accommodates multiple types of data
- Applies appropriate type of learning
- Provides probabilities regarding SA assessments (hypotheses)
- Provides evidence in support of and against SA assessments
- Allows feedback from users of the system to enhance performance
- Supports continuous, on-line learning.

Refereed Communications

N. Brannon, G. Conrad, T. Draelos, J. Seiffertt, and D. Wunsch, "Information Fusion and Situation Awareness Using ARTMAP and Partially Observable Markov Decision Processes," in *Proceedings of the 2006 IEEE World Congress on Computational Intelligence*, pp. 4025-4032, June 2006.

UGS Concept and Technology Development for Enhancing Boost Phase Detection ISR SDAC Application

79836

A. B. Doser, D. C. Craft, J. W. Giron, W. T. O'Rourke

Project Purpose

This project explored the use of networked unattended ground sensors (UGS) to enable early detection of missile launches, as well as investigate the use of precursors that indicate a launch is about to occur. These systems offer the potential for shortening detection times and giving other parts of the missile defense system more time to perform track and intercept functions. UGS may also contribute to improved robustness by using detection schemes based on phenomenology not currently in use.

FY 2006 Accomplishments

We concentrated on the networked sensor detection of precursors and launches using available data collected from previous experiments and we investigated the potential for using higher order statistics to pinpoint signal location. Despite poor data quality and time stamp accuracy, we were able to demonstrate the benefit of networked sensors for detection and classification of signals associated with launch activities. Improvements in launch detection due to networking are more difficult to prove since there were only data from two sensor locations and not a more fully populated network. However, analysis indicated that there was potential benefit from false alarm reduction and system robustness from the use of networking.

Significance

The project supports goals to develop new approaches for launch detection and event characterization for missile defense. The project yielded new algorithms and detection approaches that are also applicable in other DOE and work-for-other applications.

Large-Area Metallic Photonic Lattices for Military Applications

79838

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Project Purpose

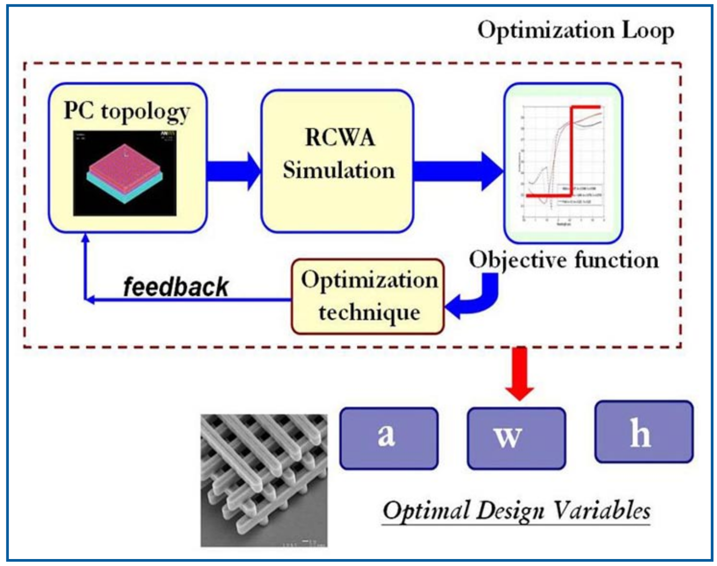
One of the "holy-grails" for many military platforms is the creation of a "chameleon skin" to control the platforms' optical and thermal characteristics. Photonic bandgap (PBG) materials are poised to have enormous impact on the observability and thermal management of a variety of military platforms and on the creation of revolutionary optical emission technologies (e.g., lighting and display). However, current PBG fabrication techniques offer limited materials choices (tungsten, aluminum) and small areas: square inch rather than the square meter areas needed in these applications.

We brought together expertise in PBG design/modeling to optimize the PBG structures to use new materials, and efforts in nanoimprinting and metal deposition/plating processes to rapidly fabricate large sheets of new PBG designs. By developing our theoretical and experimental understanding of the design and fabrication trade-offs for these materials, we will be well positioned to rapidly respond to a variety of new application opportunities.

The goal of this project is to develop the computational design tools, micro- and nanomold fabrication techniques, and material deposition processes required to demonstrate a 1 m^2 sheet of three-dimensional (3D) full-bandgap photonic crystal material.

FY 2006 Accomplishments

We continued fundamental design and fabrication studies on 3D PBG structures for the long wavelength infrared (LWIR) and visible regions. We expanded our toolset for the efficient modeling and design of photonic crystal structures by developing a new custom finite difference time domain code, and by validating a fast commercial rigorous coupled wave analysis (RCWA) code against our other simulation and experimental results. These tools enable the accurate simulation and tolerancing of known structure.



Photonic crystal structure design self-optimization tool is a user friendly intelligent iterative optimization code developed jointly by Sandia and UNM.

To enable the design of new structures for a desired optical response (reflection, transmission or absorption), we developed an optimization code. This code uses the fast RCWA calculation kernel to find a unique photonic lattice microstructure defined by the three fundamental geometrical dimensions of the logpile photonic crystal design: lattice constant (a), rod width (w) rod height (h).

We also demonstrated three new mold fabrication techniques as well as PBG structures using four new materials (Au, TiO_2, cyclotene, and polyethelene). We fabricated new PBG structures for the first time using contact lithography and evaporated gold in polyethylene, electron beam lithography and titania, and surface metal machining and gold. We invented a new hybrid nanoimprint lithography approach to provide a path for fabricating large areas of deep-submicron periodic structures with well defined cavities of waveguides.

The fabrication and test studies have enabled prediction of the issues and costs associated with scaling up these processes to 1 m^2 areas, both in the infrared and visible regions. This year's work resulted in three technical advances.

Significance

The goal of this project is to provide fundamentally new capabilities for military platforms. This development of new 3D photonic crystal materials can have a "game-changing" impact at the system level. By enabling the precise engineering of a surface's optical and thermal characteristics, the functionality of a variety of military platforms can be dramatically enhanced.

This work has furthered fundamental design investigations for 3D PBG structures for the LWIR and visible regions. In the course of demonstrating new PBG structures using four new materials (Au, TiO_2, cyclotene, and polyethelene), we have developed three new mold fabrication techniques. These new micro/nanofabrication approaches can have broad applicability to future nanoscience research and development.

This work has also deepened our understanding of the theoretical and practical design trade-offs for 3D photonic crystals and other optical microstructures. The optimization-driven PBG design code we developed has already been applied to help design selective emitters needed by our thermophotovoltaic energy production applications. Analysis of the issues and costs associated with scaling up these processes to 1 m^2 areas has identified areas of new technology research and development, including, for example, phase imprint nanolithography.

The new materials and form of these PBG structures will ensure Sandia's continued leadership in the PBG field and strengthen our intellectual property within the photonic lattice area.

Refereed Communications

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Other Communications

G. Subramania, I. Brener, P.G. Clem, I. El-Kady, J.G. Fleming, Y.-J. Lee, T.S. Luk, J.C. Verley, "Complete Three-Dimensional Gap Photonic Crystals in the Near-Infrared and Visible Wavelengths for Sensing Applications," to be published in *SPIE Proceedings*.

C.F. Schmidt, W.C. Sweatt, I. El-Kady, F.B. McCormick, D.W. Peters, S.H. Kravitz, J.C. Verley, U. Krishnamoorthy, D. Ingersoll, W.G. Yelton, G. Subramamia, J.D. Williams, "New Infrared Photonic Lattice Coating," in *Proceedings of the Novel Optical Systems Design and Optimization,* p. 628912, 2006.

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Network and Adaptive System of Systems Analysis Methodology

79839

D. J. Anderson, J. P. Eddy, J. E. Campbell, B. P. Van Leeuwen, C. R. Lawton

Project Purpose

The purpose of this project was to advance current system of systems (SoS) assessment capabilities to address identified needs. Considerable progress had been made in the development of SoS design and decision support methodologies for use in analyzing large-scale complex integrated systems such as the US Army's Future Combat Systems (FCS). While providing substantial capabilities for evaluating a complex SoS, the current methodologies do not support important issues associated with assessing SoS network-centric performance, including adaptive communications and data networks.

Networks introduce dependencies and nonlinearities that can dramatically affect SoS performance. For example, several planned FCS technologies are implemented via the network communications and network-centric operations pertaining to mission readiness and sustainment planning. These technologies and SoS performance are not feasible without network communications. Thus, treatment of network effects is crucial to realistic analysis of FCS and SoS performance.

Part of our research involved evaluating and developing network modeling approaches for possible application to the SoS and large-scale problem spaces present with thousands of integrated systems. Other efforts went into developing an understanding of the not well-defined FCS network functionality. Finally, we identified an approach, implemented it into the SoS methodology, and applied it to an FCS-like example SoS problem.

FY 2006 Accomplishments

1. We collected data and information on network design, architecture, and implementation, as well as specific FCS network information. This information includes network topology and waveform information and reliability data such as mean time between failure for specific network hardware elements.
2. We developed versions of network component (primarily radios) state models based on the collected information and data. These state models serve as functions of the individual network-enabled platforms and therefore have an effect on their operational availability.
3. We constructed a network communications example model that portrays a notional FCS force. This example problem contains platforms that are modeled after the actual FCS platforms, using notional hardware for all systems except the network. The hardware and associated data are based on the latest information supplied from the FCS program.
4. To account for dynamic and complex network modeling, we defined new concepts, such as "virtual systems," and implemented them as an enhancement to the SoS Toolkit (SoSAT), the current SoS software. A virtual system is one that has no hardware associated with it and is comprised solely of references to the functions of other platforms. The operation of network systems, which are virtual systems, is therefore defined primarily in terms of the functionality of the force platforms serving as network nodes.
5. We developed algorithmic components to aid in the definition and analysis of virtual systems and implemented the components in SoSAT. We improved the ability to define the inter-platform references that serve as the core feature of these virtual systems. Included is an algorithm to detect circular references whereby two platform functions reference one another simultaneously, resulting in an irresolvable loop of references.
6. We addressed the following challenges of SoS modeling and analysis of complex, adaptive data and communications networks:
   - Problem scale grows exponentially in complexity when the interdependencies

introduced by the data and communications network are taken into account. We addressed this problem by defining the virtual systems such that their complexity grows linearly rather than exponentially and by improving the means by which these systems are created to greatly reduce setup time and the potential for operator error.

- Network performance is highly dependent on the hardware that constitutes the nodes within the network. However, the network functions at a level of abstraction above the physical systems that form network nodes. The challenge was to model and quantify the performance of the network when the network functions at a level above the individual systems upon which it depends. To address this problem, we created the virtual systems. These systems operate like other systems except that their entire function set is comprised of references to the functions of the nonvirtual systems.

Significance

The expansion of system of systems modeling capabilities to include network modeling presents a differentiating capability, and enables Sandia to maintain its leadership role with unique capabilities and tools for analyzing complex SoS.

Our accomplishments have played a key role in obtaining increased funding to pursue additional tasks with the new capability. A new system of systems analysis project involving network modeling for the Army's Program Executive Office Ground Combat Systems has been funded, and the potential exists for more work to meet other future customer needs with the additional capabilities of the SoS and network modeling and analysis methodology.

Development and Optimization of Thermal Protection Materials for Hypersonic Vehicles

93427

P. J. Cole, R. E. Loehman, A. Ayala, D. W. Kuntz, J. A. Emerson

Project Purpose

Currently, thermal protection systems (TPS) limit the mission capabilities of hypersonic flight vehicles. We propose to develop optimized oxidation-resistant carbon-carbon composites and novel manufacturing processes that will enable vehicle performance commensurate with national goals. We envision a monolithic TPS incorporating single-structure composite concepts designed to ensure that vehicle internal temperatures remain within operating limits while minimizing TPS thickness and mass. Such a TPS solution will allow smaller vehicles to meet long-range, long-duration mission requirements.

Key challenges are development of adequate oxidation protection materials and treatments to slow oxidation kinetics, minimize ablation, and lower mismatches in coefficient of thermal expansion (CTE) that may lead to cracking of the oxidation-resistant layer. Candidate oxidation resistant materials include tailored solution-derived ceramics and siloxane-based polymeric glass precursors, each with unique benefits and challenges.

Siloxanes can form glass coatings in situ, minimizing CTE mismatches, but their use temperature is limited by glass reactions and phase transitions. Ceramics have high temperature limits but do not protect well at low temperatures and are susceptible to cracking. An oxidation protection scheme incorporating both types of materials may be synergistic.

We are investigating two approaches – infiltrating oxidation-resistant materials (self-coating) and pre-flight surface coating – and exploring combinations of nanoengineered materials and nanoparticle processing methods. Extensive high-temperature testing and materials characterization will provide an understanding of the mechanism of oxidation protection that will be used to guide further improvements.

The high-flux solar test facility (National Solar Thermal Test Facility, or NSTTF) provides a unique controlled environment for testing. The materials science and manufacturing process work will be complemented by concurrent development of innovative aerothermal modeling approaches to handle the complex chemistry effects (both surface and in-depth) of these self-coating materials and the assessment of these materials as candidate TPS for proposed long-range hypersonic cruise vehicles.

FY 2006 Accomplishments

We established a solar furnace test capability that enables improved test control and sample monitoring. The system permits surface observation via video and infrared cameras with two pyrometers to determine sample surface temperature. The capability of rapid flux ramps and controllable flux profile enabled new understanding of the response of TPS materials during the initial stages of heating. We used cross sections of test samples to identify early coating failures in samples that initially appeared undamaged. The addition of powders to siloxane-based materials to expand their performance limits gave favorable results.

We developed liquid precursor solutions for thin film coatings of ZrB_2, HfB_2, HfC, and SiC ultrahigh temperature ceramics (UHTCs). We tailored these liquid precursors for infiltration into the carbon-carbon (CC) composites to coat and protect the inner surfaces from oxidation, and for application of external coatings by dip coating. High-temperature testing of the infiltrated and coated CC showed improved oxidation resistance. We incorporated the UHTC coatings in a multilayer coating scheme that allows for self-healing in a broad temperature range. We also incorporated the liquid precursors into the composite fabrication process to produce a CC composite with ZrB_2 coated fibers.

We computationally modeled flight vehicle trajectories and heating rate profiles to guide the design of the materials experiments. We updated the existing version of the kinetic parameter estimation (KPE) code to operation because the previous code was error-ridden and would not compile. We made significant progress on developing a new, working version of KPE. The new KPE was used successfully with idealized thermogravimetric analysis data to compute Arrhenius equation coefficients, demonstrating the capability of the code to model TPS-relevant environments and materials.

Significance

New thermal protection materials and systems are needed to develop faster, longer range hypersonic vehicles, which will strengthen US national security. This project is developing optimized oxidation resistant CC composites and novel manufacturing processes that will enable the desired mission capabilities for hypersonic vehicles.

As a fundamental understanding of oxidation protection mechanisms is gained, it is being used to guide the development of new materials. In addition, existing aerothermal models for high temperature materials were inadequate, and the new aerothermal models that incorporate relevant surface and in-depth chemistry effects are being developed to improve capabilities for predicting the performance of these new material schemes, improving our design capabilities.

The key benefit of this research is the enabling of the targeted hypersonic vehicle capability. However, the new materials and processing methods that are being developed to impart oxidation resistance to CC composites will impact the broader high-temperature materials community.

Furthermore, the new, more robust aerothermal modeling tools under development permit the confident design of flight vehicles for other applications. In addition, the continued enhancement of capabilities at the NSTTF, and specifically the solar furnace, is benefiting DOE and Department of Defense programs using the facilities.

Refereed Communications

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Other Communications

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Transportable Liquid Metal Reactor System Design

93588
P. J. McDaniel, C. O. Farnum, L. C. Sanchez, V. J. Dandini

Project Purpose

The purpose of this project is to design a nuclear reactor to couple to a state-of-the-art turbocompressor to produce electricity for military applications. The system should produce ~ 200 kW of electrical power for 6-24 months and be air transportable to and from forward-operating bases.

FY 2006 Accomplishments

We completed a conceptual design for a liquid metal-cooled reactor that will drive a Rolls Royce RR250.

Significance

Design details, operational procedures, and radionuclide inventories have been identified that indicate continued evaluation of this concept should be fruitful.

Time-Frequency Enhanced Radar Processing for Foliage Penetration

93589

D. W. Harmony, J. T. Cordaro Jr.

Project Purpose

Increasing demands on remote surveillance systems to provide meaningful intelligence places a premium on extracting the most information available from sensor data. Unfortunately, imaging radars typically lose information because conventional Fourier transform processing techniques ignore the rich time-frequency correlations that exist in radar signals.

We will avoid this loss by developing through this project joint time-frequency algorithms to extract weak signals in the presence of strong multiple scattering. Although the specific problem we are considering is foliage penetration, the new algorithms are expected to have broad applicability to a wide range of radar signal processing issues. Successful completion of this project will give Sandia an unparalleled capability in remote sensing through foliage.

FY 2006 Accomplishments

We selected and developed a time-frequency algorithm for processing radar signals based on an iterative scheme that approximates the signal with a series of Gaussian pulses. The parameters for each Gaussian term are determined from a sequence of fractional Fourier transforms that find the best amplitude, duration, frequency content, and chirp rate for matching the signal at each iteration. This technique provides good time-frequency isolation by allowing each Gaussian pulse to adaptively adjust to the signal.

We identified relevant concealed target synthetic aperture radar datasets. Once we recover the datasets from the archive tapes, we will apply the new time-frequency algorithm and assess it for its ability to extract man-made target signals from foliage clutter. We also will focus on refining the algorithm and analyzing its performance in conjunction with polarization processing to amplify differences between target and foliage signatures.

Significance

The key research accomplishment this year has been the development of time-frequency processing algorithms tailored to complex radar signals. These algorithms should yield significant improvement of signal-to-clutter ratios for targets in foliage, better focusing of maneuvering targets (inverse synthetic aperture radar), and tracking of ground moving vehicles.

Secure Portal

93590
T. D. Russ, M. W. Koch

Project Purpose

To address the issue of autonomous facility access through the use of technology, we introduce the idea of a secure portal. A secure portal is a defined zone where state-of-the-art technology can be implemented to grant privileges to an individual. We will look at face and gait recognition technology to achieve this task. These technologies require less user cooperation than other biometrics, such as fingerprint, iris, and hand geometry, and thus have the most potential for flexibility in deployment.

In FY 2006 we worked to develop and evaluate face and gait recognition algorithms that can meet the high accuracy and fast computation demands of the secure portal application. The goal was to develop face and gait algorithms independently to obtain their best performance. We will begin fusion of the technologies in FY 2007.

We used two databases to support these efforts: the University of South Florida Human Identification (HumanID) gait database and the University of Notre Dame Facial Recognition Grand Challenge (FRGC) database. The HumanID gait database contains 122 subjects, 12 probe sets, and six covariates. The covariates include changes in surface type, shoe type, camera view, time, clothing, and the presence of a briefcase. The FRGC database contains more than 400 subjects and 4007 images. The data was collected over three semesters and contains some variation in facial expression. Each dataset contains the three-dimensional (3D) coordinates of the face with registered color or texture information.

The algorithm selected for face recognition is based on principal component analysis (PCA), which is extensively used for the analysis, compression, modeling, and recognition of objects because of its simplicity and ability for dimensionality reduction. PCA derives an optimal basis from a set of training examples (representing signals in class C) such that training examples can be represented as a linear combination of basis vectors.

For recognition problems, the derived basis needs good generalization performance. For instance, the basis should accurately represent new signals (not in the training set) in signal class C as a linear combination. To achieve both data accuracy and efficiency in representing new within class signals, both sufficient variability in the training set and proper correspondence of signal features (in concert with linear combination principals) are needed.

We developed and evaluated a gait recognition algorithm based on hidden Markov models (HMM). The gait matching problem is represented by an HMM in that each gait sequence is partitioned into a set of templates that are predominant over a gait cycle. Each template in this set can be considered a state in an HMM with probabilities associated with transitioning from state i to state j. Because the states represent stages within a gait cycle, they contain a defined order within a gait cycle, thus, many transition probabilities will be zero.

FY 2006 Accomplishments

Face Recognition (FR)

Our novel contribution to 3D FR using PCA is how points are arranged for input to PCA. The approach enables a good alignment of 3D points across faces while still preserving face size information important for the recognition process. This is achieved using a generic reference face model that can be scaled in 3D to best fit an input face. By scaling the reference model, opposed to the input face, proper size information of the face of interest can be encoded, and better alignment of that face to the reference can be performed.

The reference face is the foundation for a new coordinate system that provides a mapping between distinct faces to be encoded in the PCA decomposition. Input face to reference face alignment is achieved using the iterative closest point (ICP) algorithm, and correspondences are obtained using a normal search criterion. Extracted facial features are used to determine proper scaling parameters. Our FY 2006 accomplishments include:

- Developed and implemented basic 3D PCA algorithm and correspondence methods
- Improved accuracy of feature extraction used for reference face scaling
- Developed algorithms to integrate two-dimensional (2D) texture into the PCA framework
- Analyzed verification and identification performance
  - Analyzed generalization performance of PCA model
  - Analyzed compression capability of PCA model
  - Analyzed FR accuracy with variations in training set size
  - Analyzed FR accuracy using different similarity metrics
  - Analyzed FR accuracy with variations in face expression
  - Analyzed FR accuracy with variations in reference face size
  - Analyzed FR accuracy using a mean reference face
  - Analyzed FR accuracy with variations in the number of face points
  - Analyzed FR accuracy using 2D texture

Gait Recognition (GR)

The HMM used for GR determines the similarity (i.e., a matching score) between a probe image sequence and a gallery image sequence by computing the probability of observation between the two. This probability is dependent on the state transition probabilities, the initial state distribution, and the observation likelihood (the match scores between state or gallery templates and the probe gait sequence images). This model enables some consideration of both the temporal/motion (represented by the transition probabilities) and shape dynamics (represented by the observation likelihood) within a gait sequence to determine human identification.

We conducted experiments using five templates to represent a gallery gait cycle and computed a priori the transition probabilities from the gallery image sequence. Based on this method we:

- Implemented gait cycle estimation, gait template extraction, and gait frame clustering algorithms
- Implemented dynamic time warping (DTW) algorithm as an approximation to HMM, HMM with fixed transition probabilities, and HMM with learned transition probabilities
- Analyzed verification and identification capability of probes A-J of A-L of the HumanID gait database
  - Analyzed GR using different similarity scores (used in the observation likelihood)
  - Analyzed GR using DTW, fixed HMM, and learned HMM

Significance

The ability to create zones secure from unauthorized access is important to a variety of national security applications. Accurate recognition using face, gait, and other body metrics can provide confidence that the people being admitted to an area are the people who have proper authorization. The use of autonomous technology can speed throughput and reduce security costs associated with the use guard forces, thereby freeing up potential resources for other site needs.

This technology would be valuable to the US intelligence community interested in remotely monitoring increased activity around areas of interest. DOE facilities, our country’s critical energy and water infrastructures, and docks, airports, and border entry stations could also benefit.

We developed a fully automated process for 3D face recognition that achieves a 91.5 percent verification rate at 0.01 percent false alarm rate and a 97.5 percent rank one identification rate. Better performance is expected as we improve the technique, address known issues, and fuse 3D face with 2D face and gait biometrics. We showed that the performance holds up with reducing the number of points used on the input face from approximately 40,000 to 2,000 given that a large number of points is included on the reference face (i.e., 20,000) used for alignment and correspondence. The number of points required will be important when selecting a 3D sensor for this application.

In our approach, matching is extremely fast once the PCA coefficients are obtained. The current 3D PCA approach only requires the matching of an input face to a reference template. Thus, faces in a database can be encoded a priori and at match time only the input face has to be registered and encoded and can be matched with the PCA features stored in the database.

Currently, reference to input face registration and correspondence takes approximately 11 seconds in Windows XP, and PCA matching is performed at a rate of approximately 500 matches per second using 199 PCA coefficients. This means that if significant improvements can be made in software and hardware, and in the registration and correspondence stages, the technique will be feasible for a real-time or quasi real-time application.

These findings indicate that the secure portal concept is promising and will be able to impact a number of strategic missions if successful.

Refereed Communications

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T.D. Russ and C. Boehnen, “A 3D Correspondence Algorithm for 3D PCA-Based Face Recognition,” presented at the Face Recognition Grand Challenge and Iris Challenge Experiments Workshop, Arlington, VA, March 2006.

Human Perceptory Augmentation

93592

S. Buerger, K. Wojciechowski, K. A. Peterson, M. S. Bastian, R. H. Olsson, B. L. Spletzer, B. R. Rohrer, J. W. Wheeler, D. L. Kitterman

Project Purpose

Direct physical interfaces to the human nervous system could revolutionize communication between human and machine. The ability to record and stimulate from single neurons and small bunches of neurons has improved radically in recent years with the improvement of electrode fabrication technologies, and neural interfaces represent a technology area ripe for application development in the next five to ten years.

While much of the research field is focused on the replacement of lost capability through neurally controlled prosthetics, the potential for performance augmentation of intact humans has thus far been less thoroughly considered. The peripheral nervous system (PNS) holds the potential for less-invasive implantation than the brain, a consideration that is critical to the goal of eventually implementing neural interfaces in healthy subjects, as well as in amputees and victims of spinal cord injury. PNS signals are also far easier to decode than cortical signals and perhaps more intuitive to control.

In this project we are developing a peripheral nerve stimulation system designed to provide information from a suite of human-engineered sensors directly to the nervous system via a transcutaneous wireless link. While almost all laboratory neural interfaces to date use transcutaneous wires, which present a high risk of infection, we will demonstrate chronic wireless implantation, a key element of future systems for human implantation.

Custom system components include:

- stimulating electrodes
- an implantable integrated circuit (IC) to wirelessly receive digital sensor data, convert data into an analog current signal, and drive the electrodes
- decoding software to translate sensor data into appropriate peripheral nerve spike signals
- a compactly-packaged wearable microcontroller system to perform this decoding, drive the wireless interface, and completely free the subject from the need to be tethered to computers, amplifiers, or power sources.

In parallel with this technology development and in collaboration with university partners, we are pursuing animal testing of the concept of sensory augmentation through the PNS of a rat. Once the concept has been incrementally validated using conventional transcutaneous electrode technology, we will use our novel hardware to demonstrate the concept using a wireless implant.

This project will bring Sandia to the cutting edge of an important emerging area, contribute to the area of neural interfaces by doing things that have not been done before, and demonstrate the Laboratories' ability to work with top university researchers to engineer systems for applications in areas that previously have existed only in pure research settings.

If successful, this project will represent, to our knowledge, the first demonstration of sensory augmentation through the PNS of an animal, and the first application of a truly portable wireless neural interface. The portable package will facilitate new possibilities in behavioral animal research, permitting for the first time the animal to move freely over a large area with an active, fully implanted neural interface.

FY 2006 Accomplishments

We made substantial progress toward our project goal of demonstrating sensory augmentation in a rat through wireless PNS stimulation.

We formed partnerships with the University of Michigan, which will conduct animal trials, and with

NeuroNexus Technologies, which will provide custom peripheral nerve stimulating electrodes. We consulted with experts at several other institutions, including the University of New Mexico, the MIND Institute, the University of Texas, and the University of Pittsburgh.

In preparation for animal trials, we designed an incremental test plan to validate our approach and allow us to make changes and improvements as early results are obtained. Initial acute experiments on six animals at the University of Michigan demonstrated the ability to generate action potentials in the sural nerve (which is completely sensory in a rat) as well as the peroneal nerve, and allowed the Michigan team to refine its surgical procedure for the PNS.

We designed and tested flexible software to decode engineering sensor signals into variable-frequency charge-balanced spiketrains typical of peripheral nerve signals. This software will serve as a test bed for initial animal trials and will permit parameters of the stimulating signals to be changed “on the fly” for rapid optimization. We have selected and tested an ultrasonic range finder as one initial sensor test bed and derived input specifications for conversion software based on this and other sensor protocols.

We completed the design of a first-generation implantable stimulation and recording IC, including custom circuitry for digital-to-analog and analog-to-digital conversion, amplification, multiplexing, and input and output communications, and ordered the chip’s fabrication. The device includes a novel, robust digital inductive communication link that can accommodate significant variations in the coupling of the two coils due to interference from the skin. It can record a single channel of neural activity with 10-bit resolution and stream data outward. It can also stimulate one of eight sites up to currents of plus or minus 35 microamps.

We also completed the majority of the design for a second-generation implantable stimulator that will add several capabilities, most importantly permitting up to eight sites to be stimulated simultaneously. This design will be fabricated after initial testing of the first-generation device.

We initiated the design of custom probes for peripheral nerves with NeuroNexus Technologies and tested methods for attaching the probes to a printed circuit board that will hold the stimulator chip. The Michigan team is exploring ways to suture the probes into the nerves so that there is no relative motion between the probe sites and neurons as the rats behave.

Finally, we made a significant effort to educate our Sandia team on the physiology of the human and rat nervous systems, facilitating rich interaction with our collaborators in neuroscience, guiding our hypotheses on how and where to interface with the PNS, and beginning our study of the transition of this technology to eventual human use.

Significance

Neural interface technology and this project in particular stand to impact Sandia’s national security mission by radically improving warfighter performance and potentially permitting extraordinarily high-fidelity remote control of devices, vehicles and systems. Sensory augmentation through the nervous system, particularly at high bit rates, could provide a substantial asymmetric advantage for warfighters, akin to the similar advantage provided by night vision systems in the dark.

Sensors such as chemical and biological agent “sniffers” could be employed, as could arrays of laser, infrared or ultrasonic range finders to provide full-spectrum vision. The system also permits sensors to communicate wirelessly with the microcontroller mounted to the subject, such that sensors need not be carried. This could permit a neurally equipped soldier to sense data from sensors in a variety of locations (such as entrances to a building he is clearing) without having to consult a display. Perceptory augmentation could also be used to sense fellow soldier distress levels or locations.

The nervous system may also provide a promising interface for direct, high-speed man-in-the-loop control. Current human control systems (e.g., joysticks, haptic devices, steering wheels) are limited to the bandwidths of muscles, bones, and tissue, which are usually on the order of several hertz. Neural control could theoretically respond at frequencies an order of magnitude greater, limited only by conduction velocities of action potentials.

The technology developed in this project could impact the development and testing of neural interfaces in animals by permitting the animals to roam freely while the neural interface is active. Today, virtually all behavioral animal testing with neural interfaces is conducted in small behavior boxes. Neural interfaces must be wired to large amplifiers, and commutators must be used to prevent the animals from tangling themselves in wires. Completely untethered interface systems (including wireless implants as well as wirelessly interfaced microcontrollers outside the body) would remove these constraints, permitting testing of neural interface applications in realistic, open settings. Several of the university experts with whom we consulted expressed great interest in this technology as a research tool.

Finally, the treatment of amputees and victims of spinal cord injury could be advanced by this technology. In a spinal cord patient, neural signals could be transmitted across an injury by recording on one side and stimulating appropriately on the other. In amputees, remaining nerve pathways can be exploited to recover function, by recording motor neurons and feeding to robotic limbs, and by stimulating sensory neurons in response to measured signals.

By exploring augmentation, we also introduce the possibility that amputees or spinal cord patients may one day not only regain lost function, but may also gain new capabilities not possessed by a healthy subject through neural interface technology. Perhaps one day an injured soldier will voluntarily return to duty as a “neural specialist,” capable of communication and control through a direct path to his nervous system.

Advanced Hard Target Warhead

93593
R. McEntire, E. W. Klamerus, J. S. Ludwigsen, J. E. Bishop

Project Purpose

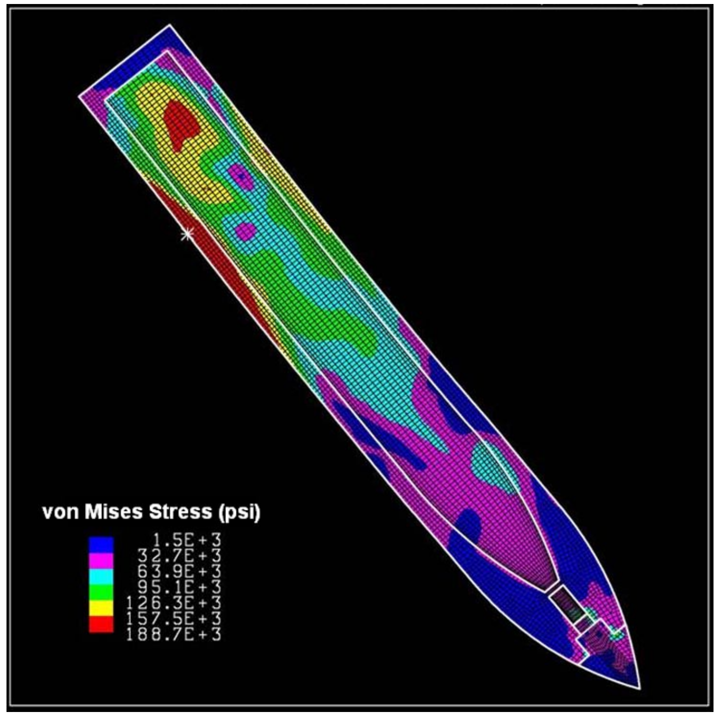
Targets of strategic and tactical importance, including command and control, communication, and storage/ production of weapons of mass destruction (WMD), are being moved to underground facilities. These facilities are constructed deeper and hardened with additional concrete to resist the penetrator weapons currently in the US conventional arsenal.

The purpose of this project is to understand the environment for the fuze and other internal electronics in an advanced, high-speed warhead design capable of defeating these targets, so that robust, reliable electronics can be built. This weapon concept will be in a class to deliver a lethal amount of explosives and will have the fuze intelligence to detonate within target voids to maximize the lethality.

The innovative approach to the proposed warhead concept is placing the fuze in alternate locations. Previous analyses and subscale testing indicated the potential benefit to fuze survival and the ability to detect voids. Additionally, the structural and functional integrity of the explosive train must be evaluated. To increase manufacturability and ease of maintenance, the study will explore designs that feature jointed penetrator cases.

The technical approach is a science-based design and analysis of the future warhead concept as a system. We will simulate the penetrator striking typical strategic targets with varying velocity, angle of attack, and angle of obliquity. The warhead analysis will include a re-entry vehicle around the penetrator. The targets will include a facility under a layer of limestone and a multilayered concrete bunker. The environments from these simulations will be available for use to explore advanced packaging concepts for protecting the fuze while simultaneously supporting the development of intelligent fuze algorithms.

To address model accuracy and relevance, we will focus on subscale testing to validate select analysis cases. This approach will yield a robust design with empirical results to validate the predicted performance and success.



Stresses in a jointed penetrator in an oblique, high-speed impact.

FY 2006 Accomplishments

In order to simulate the environment experienced by a fuze in a fuze-forward, jointed penetrator, we developed three feasible designs for penetrators in the 1000-lb class through extensive use of modeling and simulation.

We simulated the performance of various designs against representative targets such as a “cut-and-cover bunker” target, a “dry limestone” target, and various concretes. First we conducted sensitivity studies for a baseline jointed penetrator and several ballasted and unballasted variants, considering ranges of striking speeds, angles of obliquity, and angles of attack, using PENCURV. These studies guided us in selecting basic penetrator design parameters, such as the length-to-diameter ratio.

We then developed three jointed penetrator designs, with the fuze in various alternate locations. We simulated high-speed impacts of these in targets of varying penetrability and varying angles of obliquity and attack using the Presto transient solid dynamics code with spherical cavity expansion (Presto/SCE). All three designs survived impacts over ranges of conditions that depend on the impact speed, but had differing penetration performance as well as differing manufacturability and maintainability features. One of the designs, for example, is probably more difficult to manufacture but easier to maintain.

The Presto/SCE simulations show where peak axial and lateral accelerations and maximum stresses and strains occur in the penetrator. This is important for designing both a penetrator joint and a case that will survive the penetration event. The simulations also provided bounds on the range of impact angles of obliquity and attack for which the penetrator path in the target is stable. We conducted two studies of path stability using Presto/SCE. One was based on the path deviation for a generic penetrator to indicate stability. The second used the three jointed penetrator designs and used penetrator deformation to indicate path stability. As the impact speed increases, the ranges for path stability decrease. The simulations provide more precise estimates for the ranges.

The fuze environment simulations provided detailed predictions of the mechanical environment for the fuze. The stresses and strains on the fuze surrogate in the simulation can be used by fuze designers to develop more robust electronics and to develop packaging to mitigate the effects of the mechanical environment on the electronics. This information will also be useful for developing acceleration-based intelligent fuzes. The simulations also showed that the force exerted by the high explosive on the fuze during the impact may play a significant role in the reliability of the fuze.

A caveat regarding the simulations with PENCURV and Presto/SCE is that neither code has been validated at the high impact speeds we considered in our studies. Subscale tests next year will help determine the validity of these simulations.

Significance

The goal of this project is to provide a more fundamental understanding of the environment seen by electronics in penetrators in very high-speed impacts. Understanding this environment will enable the design of more robust fuzes for both conventional and nuclear applications as well as more robust data recorders. Fuzes, especially penetrator fuzes, promise to be a significant business area for Sandia.

Understanding the environment also supports the development of intelligent fuze algorithms, algorithms for detecting voids. The ability to reliably detect voids and detonate in them can be used to enhance weapon lethality. The work in this project has particular relevance to the "Intelligent Fuzing for Hard Target Defeat" LDRD project (79869) and the "Void Sensor for Penetrators" LDRD project (93600).

This project also provides fundamental understanding of jointed penetrator designs and their performance limits in high-speed impacts well beyond current practice. This work therefore supports Sandia's significant technology base in penetrator technology.

The aggressive and early use of modeling and simulation in penetrator design, if validated by the testing planned for the second year of the project, will enable Sandia to move away from a "build and test" methodology toward a "simulate and test" methodology with concomitant improvements in quality and cost effectiveness. This is true not only of the penetrator but also for the fuze or data recording electronics package that rides along during the test.

The knowledge gained from simulations of high-speed impacts can guide future investments in penetrator joint and case design, guidance and control technology, shock-hardened electronics design, electronics packaging design, and intelligent fuze algorithms development. These areas in turn impact such ongoing Sandia activities as the Defense Threat Reduction Agency Hard Target Defeat program, the Department of Defense/DOE Munitions Memorandum of Understanding, and the Tactical Missile System-Penetrator (TACMS-P) program.

Information System Situational Awareness

93594
B. P. Van Leeuwen, J. T. Michalski

Project Purpose

The purpose of this project is to develop an analysis tool that combines detectable attributes and events to create a normal system operation profile. Once a system normal-operation profile is known, it is compared to a current-operation profile to identify system malfunction or unintended operation resulting from compromise. Since the system operation profile provides insights into system purpose, capability, and state, the tool supports creating system protection techniques to hide its profile from adversaries.

This research differs from computer network intrusion detection and firewall technology by conveying system-wide operation characteristics and not focusing on specific application-based signatures. The tool greatly advances an analyst's ability to characterize information system composition, operation, function, and state from detectable attributes and events.

The tool will function as a system, incorporating the strengths of computers and computer networks for data collection, filtering, processing, and visual rendering with the strengths of human analysts in recognizing patterns, roles, and relationships. Analysts will dynamically interact with the tool to integrate situational information and obtain characteristics of the information system based on detected attributes and events.

The research advances the:

- collection and merging of information system situational awareness (ISSA) data using passive and active methods
- analysis of ISSA data to gain insights into the system characteristics
- information presentation and attribute estimation, resulting in a system-wide common operating picture.

FY 2006 Accomplishments

We selected a sensor network scenario as our initial target information system to which the developed capability will be applied. This selection allows us to focus our attribute and event collection to those applicable to a sensor system. We identified several categories of data that can support information system situational awareness estimation. Categories include data types that are collected directly from the system's supporting communication network and data types that are collected from detectable physical attributes of the system.

We identified and are extending a framework that matches models of information systems to detected attributes and events of the system. This framework employs a rule-based system using CLIPS (C Language Integrated Production System). CLIPS is an expert system tool that provides an environment for the construction of models and object-based expert systems. These models and systems are used to match detected attributes, detected events, and asserted attributes to known patterns identified in the models. The expert system tool employs an inference engine that matches facts against patterns and determines which models are applicable.

We began investigating techniques to present the distilled and processed data to the analyst. Developed analysis techniques distill the collected data for final characterization by the analyst. The means of furthering the process of characterization and situational understanding is done with visualization techniques to analyze the patterns identified in the initial analysis and distillation of the collected data. We are employing visualization techniques developed at Sandia that generate graphical images that support a rapid interpretation by the analyst to create and maintain an information system common operating picture.

Significance

Protecting the information systems that support our nation's various critical infrastructures is paramount to national security. Our research will support this important security need by developing a tool to provide an enhanced understanding of information systems, assessing their security, and determining their operational state.

Our accomplishments support Sandia's mission in securing our homeland. Many of the systems under development at Sandia to provide physical monitoring and force protection depend on information systems that process data obtained from a variety of sensors distributed over a physical area.

Data from the distributed sensor system is transmitted via a network to various data collection centers that process the data. Adversaries will attempt to disrupt these data networks and information systems to compromise the protection system. Thus methods and tools must be developed to protect these networks and systems from adversarial attack and/or disruption. Additionally if an attack on the network or information cannot be prevented, a means to immediately notify the system operator that an attack has occurred must be available.

This analysis tool will play a key role in determining the overall status of critical-application protection systems since information plays an important and increasing role in applications such as force protection and physical monitoring. Information systems, such as sensor systems, are a key technology to provide solutions to a broad set of customers and protective applications.

The operation state of sensor systems and their supporting data-communication networks are opaque since malfunctions are typically detected only when measurable communications errors occur. Abnormal system operations are difficult to detect when communication errors do not occur and can result in adversarial-induced system malfunctions going undetected.

Human Performance Modeling for System of Systems Analytics

93595
C. R. Lawton, J. E. Campbell, J. C. Forsythe, D. P. Miller

Project Purpose

The goal of this project is to develop a methodology and toolset for human performance modeling (HPM) within the context of a large-scale military system of systems (SoS) simulation. While current simulations account for failures of equipment and platforms over time, humans are modeled as perfect operators.

Continuous and sustained military operations produce deficits in soldiers' cognitive performance abilities that can result in inefficiencies, errors, and even death (e.g., friendly fire). Accordingly, similar deficits in the ability of existing models to predict human performance and assess accident risk are evident.

The thrust of this project is to develop performance prediction models, within a state modeling (SM) environment, that can be used to model and predict performance of soldiers engaged in virtually all aspects of military operations, including logistics and support activities. Sandia developed and uses SM concepts to model large complex SoS, a unique and state-of-the-art capability.

However, efforts to date have focused primarily on military hardware systems, analyzing performance attributes such as mobility, lethality, availability, and so on. In this project, humans/soldiers will be represented as objects within the SM environment. The performance prediction models will then be used to model state changes of soldiers and optimize performance management throughout continuous and sustained military operations.

FY 2006 Accomplishments

We researched suitable existing models of human performance within the context of military combat simulation. Using sources such as the AGARD Designer's Guide to Human Performance Modeling, those most applicable to SoS analytics appear to be task network models, cognitive and knowledge-based models, and human-reliability models. Each of these model types is focused on detailed modeling of a single person in a certain environment, so the challenge is to draw from and extend these potential modeling techniques to a large-scale environment of approximately 3500 soldiers.

We evaluated three well-known cognitive models: Soar, a cognitive modeling architecture developed at the University of Michigan; ACT-R, a cognitive modeling framework developed at Carnegie Mellon University; and Scream, Sandia's cognitive modeling framework. Because each of these frameworks requires personal data to populate the model and considerable computing resources, we developed a hybrid approach, whereby the decision-making officers of the simulation (nine percent) would be represented by a sophisticated cognitive model, and the remainder of the forces (91 percent) would employ a more simplified approach.

Our current proposed architecture in this regard is to use Scream, modeling in high detail nine percent of the population. We developed a human-reliability modeling approach for the bulk (91 percent) of the forces that assumes reliable performance in the absence of detrimental performance-shaping factors (PSFs), such as fatigue, injury, stress, emotions, inadequate training, and adverse environmental conditions.

We consulted the combat PSF literature and identified 19 internal PSFs (e.g., cognitive throughput and hunger); 12 external PSFs (e.g., terrain and vibration); and 16 task-related PSFs (e.g., fatigue, stress, and injury) as being important to combat activities. We summarized and documented the known effects of each of the 47 PSFs on combat performance. We developed a matrix with 47 rows (PSFs), and

22 columns representing common military tasks taken from the FCS Brigade Combat Team Military Operational Classification Structure. For each cell in the 1034-cell matrix, the effect of the PSF was coded as not having any effect, mildly detrimental, moderately detrimental, or severely detrimental, according to the literature.

Preliminary results suggest that the most detrimental PSFs include inadequate training, physical injury, high stress, physical fatigue, and cognitive fatigue. The next step will be to select a subset of the 47 PSFs and develop quantitative models to be included in the SoS analytics simulations. We plan to acquire veterans' estimates of combinations of PSFs on selected tasks to allow for more accurate modeling of the effects of multiple factors in complex simulated environments.

Significance

Successfully integrating HPM into the SM framework would provide a consistent modeling framework capable of capturing both equipment and human performance. SM has proven successful in realistically capturing the behavior and dependencies among more than a thousand systems in a SoS. Broadening the approach to include human performance may be the only approach to modeling cognitive effectiveness of large-scale SoS problems.

This work will benefit Army-funded tasks at Sandia that employ SoS modeling capability, including countermine, network vulnerability, modeling and simulation support, and military operations in urban terrain analytics. In addition, the capability could be applicable to Navy, Air Force, and Joint Strike Fighter programs.

This project will make a foundational contribution to the theory and practice of SoS design and decision making and to transitioning such theory into applications. Integrating HPM into Sandia's SoS methodology will open new avenues of application research and development for other Sandia simulation technologies.

Consistent with our national security mission, this project addresses SoS modeling and simulation for evaluation of integrated systems inherent in the Army's unit of action program, overall Army transformation, and national security systems. Development of these capabilities will enable Sandia to maintain a leading-edge SoS assessment capability and become a preferred supplier for system sustainment tools. In addition, the project addresses areas of need identified by the DoD (e.g., modeling of human performance and behavior).

Enabling Immersive Simulation for Complex Adaptive Systems Analysis and Training

93596
M. R. Glickman, P. G. Xavier, E. M. Raybourn, D. H. Hart

Project Purpose

The purpose of this project is to leverage our unique high-fidelity physical and cognitive modeling technology to create or contribute to immersive, human-in-the-loop simulations. The goals of such simulations may range from training to analysis. We do not seek to create this capability from scratch; we seek to employ existing technology wherever possible.

FY 2006 Accomplishments

We conducted background research and hands-on exploration to devise a comprehensive engineering plan. Background research included literature review, extensive discussions with technical experts (both internal and external to Sandia) as well as potential internal users of the technology we are producing, and conference attendance. Hands-on explorations included:

- Conducting experiments with the Umbra modular simulation framework to determine the feasibility and utility of performing graphical rendering within a separate thread of execution from the rest of the simulation
- Adding sensorimotor infrastructure to the Umbra framework to enrich the ability of avatars with rich behavioral models to perceive and interact with each other and the world.
- Demonstrating the ability of Umbra to control entities in the popular commercial serious game platform, Unreal Tournament, via a network socket interface
- Building an exploratory prototype of a software tool to help with the avatar design process.

In addition, in collaboration with a separate Sandia project, we designed a first-person interface to an analytic battle simulation. The resulting interface confirmed two project hypotheses – proving very useful for the simulation developers, while also highlighting the benefits of the additional capabilities we are developing.

As a result of our investigations, we identified three key areas of need: performance, composition tools, and character behavior. Based upon our research and explorations, we devised a comprehensive engineering plan for FY 2007/2008 that encompasses three parallel efforts targeted at meeting the identified needs.

Significance

We aim to provide a common platform that will enable both high-fidelity simulation and an immersive human interface. This platform will include content-creation tools, particularly for developing human models with complex behaviors. We expect these innovations to provide a significant increase in the accuracy of so-called “serious games” for training and analysis as well as a highly significant reduction in the time and effort to create such applications. More broadly, this project is increasing Sandia’s technical expertise in a transformational technology with benefits to national security.

Pulsed Power Integration for Advanced Electric Weapons Platform

93597
M. S. Aubuchon

Project Purpose

The purpose of this project is to address the integration of pulsed power systems for electromagnetic (EM) guns and weapons onto military platforms. Drawing on expertise from Sandia, industry, and the military, we will evaluate the problem for military platforms, including the Army Future Combat System (FCS) and Navy DD(X); create mitigation strategies; and develop tools and methodologies for controlling compatibility issues for EM weapon systems.

Electric guns and weapons are currently under extensive development by the Department of Defense (DoD). Development of hybrid electric platforms for the DoD (DD(X) and FCS) provides the on-board electric power needed for these weapons. Mobile military systems are inevitably constrained in size, leading to strong coupling between the pulsed electric system and the weapon, with deleterious effects. A primary concern is feedback from the pulsed power system into sensor electronics and power control systems in the platform leading to large power transients and disruptions.

We will investigate a compact pulsed power supply, including capacitors, rotating store/pulsed alternators, and solid-state components for coilgun power. Without a high energy density storage system, no pulsed power supply will fit onto a reasonably sized platform. Thus far, there has been no systematic investigation of this problem, and the system implications have not been thoroughly evaluated, which makes this work an important step towards fielding EM weapons.

FY 2006 Accomplishments

- We investigated solid-state components for compact pulsed power supply.
- We identified potential issues related to using solid-state devices in pulsed power supplies for coilgun launcher.
- We developed a baseline compact power supply.
- We identified technological issues for driving a coilgun with rotating store.

Significance

The outcome of this project is important for the success of EM weapon technology, which provides greater capability and solutions to potential problems facing future military platforms. It is also relevant to Sandia's mission to develop enabling military technology. We have already demonstrated that the technology works; to be successful, we need to transition the technology out of the laboratory and into the field. This transition cannot take place until we thoroughly investigate the impact of these new technologies on military platform systems.

Future military weapon system capabilities will be based on hybrid electric drive propulsion, high power capability, and exploitation of electric guns and directed energy weapons. This project will put Sandia at the forefront of these new weapons applications. Our results will provide information about the integration of EM weapons into the DD(X) and FCS platforms and provide a foundation to assess the integration with other platforms. These integration issues will become increasingly important as the military moves to all-electric vehicles.

Our results will benefit the Army FCS weapons platform, the Navy DD(X)-CG(X) long-range fire support and direct-fire development, and EM missile launch for the Navy and Ballistic Missile Defense. The National Aeronautics and Space Administration is also interested in development of EM for launch-to-space programs.

Terahertz Diagnostics for Impact-Flash Spectroscopy on Light-Gas Gun

93598

M. C. Wanke, T. F. Thornhill III, W. D. Reinhart, E. A. Shaner, M. Lee, W. J. Zubrzycki, C. T. Fuller, A. D. Grine, R. J. Lawrence Jr.

Project Purpose

This project expands the diagnostics for dynamic spectroscopy at our gas-gun facility from the visible and infrared into the terahertz region of the spectrum; these capabilities will be used for missile-defense-related studies. We recently demonstrated that impact-flash spectro-scopy is a credible technology for near-real-time missile-defense kill assessment and target typing – long-standing, unsolved problems within the missile-defense community. Our gas guns constitute a unique test bed that provides well-controlled experi-mental environments that match operational conditions relevant to missile-defense engagements. Extending our impact-flash spectral measurements into the terahertz regime will allow us to perform tests and analysis in this new spectral regime, and it will complement our current capabilities in the visible and infrared.

Terahertz spectroscopy may offer significant advantages for missile-defense applications. In particular, the terahertz region is less crowded, and much colder emissions can be seen. Terahertz spectra relate to molecular motions, some of which have been cata-loged for operational materials of interest. The combined capabilities would offer a much broader range of material identification possibilities than achievable with the atomic measurements provided by visible and infrared spectroscopy alone.

Our effort will seek to develop new methods for terahertz diagnostics on gas guns. It will also provide new measurement and recording systems appropriate for terahertz investigations in these dynamic environ-ments. Successful implementation will provide a unique laboratory-scale testing platform for important missile-defense applications.

FY 2006 Accomplishments

We measured the short duration terahertz emission from the by-products of a high-velocity impact of a Lexan slug into Comp-B using a two-stage gas gun. This revolutionary measurement had never been tried before and pushed our best terahertz technology to its limits.

We observed short pulses of terahertz emission using three separate detectors. A Nb hot-electron bolometer allowed us to capture the temporal variations of the pulse. The Si bolometer demonstrated that the tera-hertz emission was actually large as it saturated the detector. Given the large signal, we were also able to test a state-of-the-art research detector with a very small detection bandwidth, which also saw a signal, even though modeling efforts earlier in the project suggested we would not have enough emission to observe.

Unfortunately, our terahertz technology could not spectrally resolve the emission lines of particular gasses, which is desired to enable molecular identification of gasses in the debris cloud, but the large signals on the Si bolometer suggest that we are not far from reaching that goal. A differential terahertz spectroscopy tool that will be completed soon for another project should have the ability to identify specific lines and may be tested on future projects.

Significance

Our results have started us down a path toward imple-mentation of this dynamic diagnostic capability to provide a laboratory-scale test bed for investigating dynamic, time-resolved, terahertz spectroscopy. Longer wavelengths have not been previously studied under the dynamic conditions associated with high-speed impact, and may be used to solve long-standing issues of missile-defense applications such as discrimination, kill assessment, and target typing. This new and unique test bed will provide the only facility for investigating the full range of parameters associated with various missile-defense engagement scenarios.

Void Sensor for Penetrators

93600
P. D. Coleman, R. A. Bates, A. Mar, J. E. Lucero, C. O. Landron

Project Purpose

It is difficult to defeat hard and deeply buried targets. Penetrator weapons such as the BLU109 are available with accelerometer based sensors and backup time delay triggers. An alternative would be a noninertial sensor that allows for real-time fusing decisions.

The purpose of this project is to develop radio frequency (RF) and optical sensors for hard-target penetrating munitions. Sensors probe the dielectric properties (as opposed to mechanical properties) of the medium that surrounds the penetrator in order to detect voids (underground rooms or spaces). Our RF system uses a continuous wave radar approach to monitor the input match of the antenna-sensor and to determine if the surrounding medium is mostly air or solid; it then uses that information to make a fuzing decision. The optical system uses high-g tolerant, high-frequency vertical cavity surface emitting lasers (VCSELs) and resonant cavity photodetectors and is based on detecting reflected light from the ambient environment.

FY 2006 Accomplishments

- We developed a detailed statement of work and test plan that included taking a staged approach to build and test prototypes of increasing capability.
- We built and tested an RF version of the first prototype. We completed the design and fabricated both the circuit board and the patch antenna. After construction, we characterized the hardware in the lab and at a mound test facility. The circuit operated successfully in almost all scenarios.
- We mated an optical front end to the signal conditioning circuitry and tested the configuration.

Significance

Fuzing based on a dynamic knowledge of a penetrator's surroundings would greatly increase the effectiveness of penetrators. The technology for RF/optical-based, real-time media characterization has several potential DOE Defense Program and Department of Defense applications, including advanced fuzing for future penetrating nuclear weapons with very low yields, and sensing elements in possible future weapon security systems.

High Energy Density for Electric Weapons Platforms

93601

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Project Purpose

The Department of Defense (DoD) vision for future electric weapon platforms includes the development of electrical pulsers that exceed state-of-the-art in energy density by a factor of ten or more. Ceramics or ceramic composite materials have the properties consistent with those objectives. The major challenges to overcome are quality control in large samples, testing at high voltages (~ 0.5 MV), and suitable nanosecond diagnostics.

Our goal was to reengineer current manufacturing techniques to produce bulk samples of newly developed materials that may be suitable for DoD application. We performed testing/evaluation of the materials in the parameter space of the DoD applications, which will lead to the development of a demonstration prototype. With this project we aim to create a breakthrough technology that will enable unique Sandia concepts to solve a very broad spectrum of national security problems. Previous tests confirmed that > 1 J/cc is achievable.

FY 2006 Accomplishments

We used simple single dielectric layer disc capacitors comparable to commercial high-voltage (HV) ceramic capacitors in process variable studies for the ceramic loaded polymer material technology. These capacitors operated at > 50 kV, are ~ 1 nf, and tested as high as 2.25 J/cc, compared to 0.02 J/cc for the commercial units. This accomplishment exceeds our energy density goal for FY 2006.

One of our goals was to improve the state of the art on gigawatt class HV capacitors. The state of the art with this class of capacitor is based on paper, foil, film, and oil, and yields energy densities of ~ 0.1 J/cc. The single layer disc capacitors will not scale easily to this class of capacitor, so we transferred the information gained from the process variable tests to building HV multilayer devices. This more complex geometry introduces a number of complicating factors. Our current best effort with this class of capacitor is ~ 0.6 J/cc (a significant improvement).

We ran a wide variety of process variable tests on nanopowder ceramic-loaded polymer with simple single layer finished 2" disc capacitors. The energy density for the insulating material and the finished capacitor was calculated using a voltage derating of 25 percent from voltage breakdown strength (VBS) and a 75 percent packing fraction for realistic estimates that might be applied to the more complex multilayer structures required for gigawatt class capacitors. Improvements in the manufacturing process have given results which would indicate that > 1 J/cc will be achievable for the more complex capacitors.

Building the more complex capacitors involved a number of complicating factors. The structure required to hold the conductors for the multilayer design in place while minimizing wasted packing fraction gives an HV breakdown path within the structure. Several design iterations addressed this issue. These design changes included conducting spacers, hollow dielectric spacers, and ceramic load polymer filled dielectric spacers. We achieved the best results using ceramic-loaded polymer-filled dielectric spacers.

The casting process, which involved drawing thick slurries of ceramic loaded polymer resin into the multilayer structure without introducing voids, proved to be problematic. We designed and built a vacuum filling chamber that allowed the vacuum level to be controlled to near a point slightly higher than vaporization pressure of the constituents of the resin. This chamber also had vibration capability to aid in bubble removal as well as defeat problems of even flow through out the structure. We saw a factor-of-six increase in the average VBS of these complex structures as we advanced our ability to control the process.

Significance

Extending the state of the art in HV and high-current capacitors is vital for future DoD missions. Future combat systems, all electric ships, and practically every conceivable directed energy application either requires or would greatly benefit from the energy density gains this technology promises. This work has attracted interest from the Army and two industrial defense partners beginning projects in FY 2007 that may leverage this technology. As the technology matures and the processing costs begin to go down, this will impact facilities such as ZR by reducing the overall size. This technology positions Sandia for a lead role in weaponization efforts in directed energy.

Tracking Moving People with Radar Using High-Range-Resolution and Clutter Attenuation

93602
J. T. Cordaro Jr, B. L. Burns, S. E. Allen

Project Purpose

There is current interest in monitoring large areas to detect the movement of vehicles and people. Ground moving target indicator (GMTI) is a radar mode that can scan an area and detect moving targets. GMTI radar can be used alone to track movement or as a queuing device for alerting other sensors. Previous measurements at Sandia have shown that walking people have average radar cross sections (RCS) on the order of 10 dBsm and velocities of 1 m/s or less. The purpose of this project is to investigate the possibility of enhancing GMTI radar to detect slow, low-RCS, targets such as people and small vehicles.

To detect slow, low-RCS targets we will use a combination of high-range resolution (HRR) and clutter attenuation. Slow targets are masked by ordinary ground clutter. For distributed clutter, the target-to-clutter ratio can be improved by HRR. Large-RCS vehicles, such as trucks, can be detected in weak ground clutter. Low-RCS targets, such as people, are still not normally visible at HRR, even in mild desert clutter. Clutter attenuation is a known technique that attempts to null ground clutter using multiple antenna phase centers. We have not previously combined these two techniques on the same radar.

An initial part of our work will look at the system trades involving the number of antenna phase centers. Specifically, we will compare detection and angle measurement performance for two- and three-phase-center antenna designs. Angle measurement is important, because moving targets cannot be located based only on range and Doppler frequency measurements. This is because the measured Doppler frequency depends on the target azimuth position as well as its unknown velocity.

We will consider standard amplitude-comparison and phase-comparison monopulse antennas, as well as various three-phase-center linear arrays. It is expected that this work will involve radar system analysis combined with development help from antenna designers. Because HRR requires the antenna to have high radio frequency bandwidth, some antenna designs we have used in the past will not be suitable.

The level of clutter attenuation that can be obtained is affected by antenna characteristics. Departure of practical antennas from ideal characteristics will reduce the level of attenuation. We expect to study the effects of various antenna errors.

There is a possibility that a radar designed for another purpose but one similar to what we are considering, will be available for limited tests. If so, we should be able to get more data on typical RCS values and motion characteristics for people walking on the ground.

FY 2006 Accomplishments

Our goals for FY 2006 were to look at system trades between different antenna types useful for clutter attenuation and, if possible, obtain flight-test data for slow, low-RCS targets.

In thinking about system trades, we kept in mind that a GMTI radar needs to detect targets in the clutter as well as targets moving fast enough to be out of the clutter. We collected data from a Sandia radar. The radar was not designed for our project, but we were able to get useful RCS data for walking people. Additionally we learned how important it is to minimize the cross-polarization antenna response.

Significance

Our key accomplishment was to determine the system trades involved in adapting and extending GMTI radar to the problem of detecting and tracking people and other slow, low-RCS targets. The particular interest has been to constrain the problem to a radar operating on a relatively slow unmanned aerial vehicle. This constraint limits antenna size, which has a number of negative consequences, but it still allows a capable and relatively inexpensive system.

In FY 2007 we will develop an antenna design. We will then look for outside funding to build and test a working radar. Much of the effort is leveraged on our existing synthetic aperture radar (SAR) hardware, software, and infrastructure. An integrated product would provide both SAR and GMTI radar modes.

This work will benefit Sandia missions related to intelligence, surveillance, and reconnaissance and persistent global awareness.

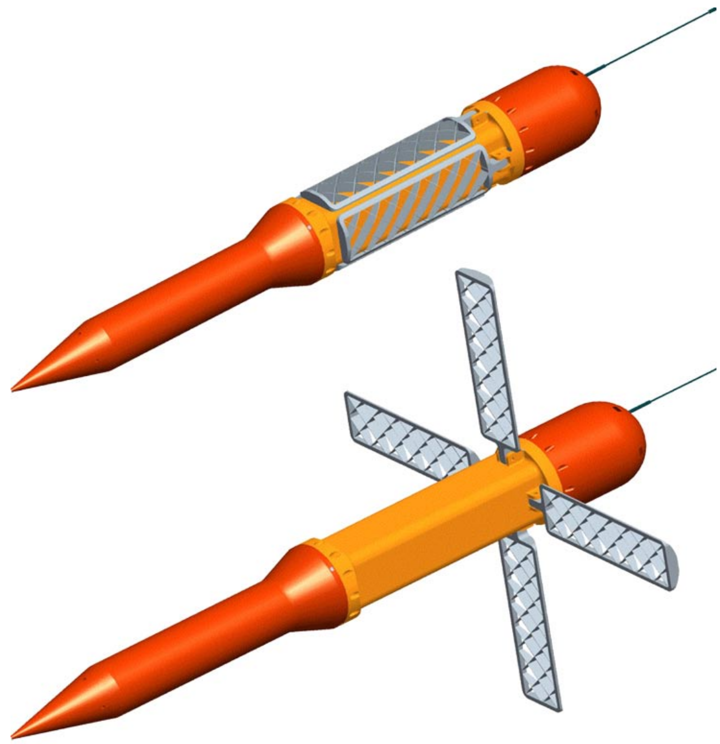
Miniature Air-Deliverable Guided Sensor System

93603
R. J. Fogler, J. T. Foster, M. S. Howard, D. E. Gallegos, M. W. Kniskern, B. P. Danowsky, J. R. Phelan, J. R. Van Houten

Project Purpose

Unattended ground sensors (UGS) are needed to provide accurate and timely intelligence for own-force protection, strike planning, and bomb damage assessment. Hand emplacement is precise but places personnel at risk, and the deployment timeline is often inadequate for transitory targets. Current air-deliverable UGS are unguided, which limits their utility where precise emplacement is needed. Existing earth-penetrating UGS are relatively large, weighing tens of pounds, which restricts their numbers on delivery platforms.

We are developing a detailed preliminary design for a miniature, guided air-deliverable sensor package with improved delivery location accuracy and reduced weight and size compared to existing systems. We focused our efforts on designing a guided miniature air-deliverable dart (GMADD) penetrator for deployment in remote areas.



CAD drawing of baseline GMADD penetrator

A number of technical challenges exist for the GMADD dart design:

- Lighter weight requires a very small diameter to ensure adequate penetration into hard soils, but a smaller diameter makes the internal packaging of power systems and electronic components more challenging.
- Developing an aerodynamically stable dart with reduced physical size and weight is complicated by the need for quicker reactions to perturbations in air flow in descent from platform release, the possibility of a reduced length-to-diameter ratio, and the difficulty of maintaining a forward center of gravity since internal component placement options are more limited.
- Achieving near-zero angle of attack at impact from a semi-ballistic guided trajectory needed for accurate placement is challenging.
- Maintaining full mission capability in a small form factor requires improvements in stored energy density and/or reduced power consumption compared to existing systems.

The design of an air-deliverable sensor system involves a number of complex tradeoffs. Since changes in one design aspect can have profound effect on others, an iterative approach is necessary. We performed a baseline design the first year to enable initial aerodynamic and penetration analysis and to provide a framework for evaluating packaging options. We modeled sensing, processing, and communication parameters after those of a recently completed unguided sensor dart project, with the additional objectives that GMADD: (1) be miniaturized to fit three units within the existing single-dart air-delivery platform, and (2) achieve a delivery circular error probability (CEP) approaching one fifth that of the existing dart.

FY 2006 Accomplishments

To establish initial foundation for penetration and aerodynamic analyses, we developed an early baseline GMADD physical dart design using somewhat preliminary miniaturization assumptions. For the baseline design, the controller and analog electronics were reduced 2.2X and 4.3X in volume and the radio volume was reduced 2X. This represents one fourth the volume and weight of the Defense Advanced Research Projects Agency's (DARPA) Sensor Dart, permitting three darts to fit on the Sensor Dart glider platform. One of the darts must face backwards to fit within the desired volume. We are planning further miniaturization that will allow three or more darts to fit facing forward.

We performed simulations with the baseline design demonstrating proper penetration in soils with S-numbers in the range 3 to 12, for an impact velocity of 100 m/s. Guidance will be accomplished using four servo-controlled grid fins, a low-cost solid-state six-degree-of-freedom inertial navigation system module, and a global positioning system receiver. We determined the grid fin size for the baseline design using an aerodynamic drag model and the assumed terminal velocity. We verified aerodynamic stability through simulation using wind tunnel data from the DARPA Sensor Dart and grid fin data from the MOAB (massive ordnance air blast) design.

Initial indications are that GMADD will be able to steer a cross-range distance of up to 965 feet from straight ballistic trajectory from a release altitude of 3000 feet above ground level in calm wind conditions.

Significance

In response to emerging national security threats, Sandia is seeking advanced solutions to defend the nation against weapons of mass destruction, defeat hardened deeply buried targets (HDBT), combat terrorism, and support related missions. Areas of emphasis include characterization of deeply buried and or time critical targets, technologies to support remote emplacement of tactical sensor systems, weapon borne sensing systems, and networked sensing systems for characterization and defeat in complex terrain or urban environments. In addition, innovative sensor technologies and microsystems-enabled sensor systems are sought for force protection, urban operations, battle damage assessment, and other important national security areas. This project addresses technical challenges and advances capability in many of these emphasis areas.

We are developing a miniature air-deliverable guided sensor system that will be capable of monitoring and characterizing HDBT facilities through the reception of seismic, acoustic, and electromagnetic emissions generated by the facility. It will also be capable of providing post-strike battle damage assessment via activity monitoring and analysis of weapon detonation signatures. The reduced size and weight of these air-deliverable units will improve covertness and permit larger quantities to be delivered from limited airborne assets. A reduced delivery CEP will improve the likelihood of successful air-delivery into tight spaces and/or in closer proximity to targets of interest.

With the addition of autonomous internal guidance, we will establish a new performance bar in the delivery accuracy of tactical sensor systems. Better delivery accuracy will not only improve effectiveness in facility monitoring, but also in remote surveillance missions for force protection where unguided air-delivery is not feasible, such as along tree-lined roadways and small clearings in forested areas.

Nonmechanical Zoom Using Active Optics for Night Vision Goggles

93604
D. V. Wick

Project Purpose

Night vision has revolutionized our ability to identify and defeat threats. As the technology improves, the military has increasingly used night vision goggles (NVGs) to gain a tactical advantage. In spite of the advances, however, NVGs remain heavy and cumbersome and provide only a limited field-of-view (FOV). Soldiers who use NVGs ask for these improvements: a variable FOV, increased battery life, and reduced weight and size. And, the Army recognizes that the current NVG capability of a 40-degree FOV is insufficient.

The purpose of this project was to assess the potential for using nonmechanical zoom, recently patented, to address the inherent deficiencies in NVGs. Our focus was to study the potential for building compact, lightweight, low-power NVGs that improve a soldier's ability to maintain situational awareness without sacrificing higher resolution for threat identification. Such improvements would be useful for field soldiers and improve the ability of security forces to maintain installation security.

FY 2006 Accomplishments

- We assessed the design tradespace for the night vision application. Unfortunately, currently available devices (liquid crystal lenses and spatial light modulators) prohibit the compactness necessary for NVGs. However, as better devices become available, nonmechanical zoom for NVGs is possible.
- We designed a 4.65X nonmechanical zoom system using the lens design program Zemax.
- We demonstrated a 4.65X nonmechanical zoom using a liquid crystal spatial light modulator and a liquid crystal lens. We also showed the ability to magnify objects off-axis. With conventional zoom, the object to be magnified must be on-axis.
- We developed a concept for increasing the diffraction-limited spectral bandwidth of NVGs using active optics and a nonmechanical spectral filter. We submitted a patent application on this concept.

Significance

While currently available devices are insufficient for the NVG application, we successfully demonstrated a relatively compact 4.65X zoom system that could magnify objects off-axis. This technology will be useful in other applications, such as small unmanned aerial vehicles, unattended ground sensors, and military rifle scopes. We also believe that liquid crystal and electrowetting devices will continue to improve. In the next two to five years, we believe those devices will have sufficient dynamic range for integration into NVGs.

In addition, the enhanced spectral bandwidth development has applications in not only night vision, but also for spectral discrimination. The ability to discriminate multiple spectral bands from a nonmechanical, compact imaging system has applications for chemical/biological, improvised explosives devices, and nuclear by-product detection.

Other Communications

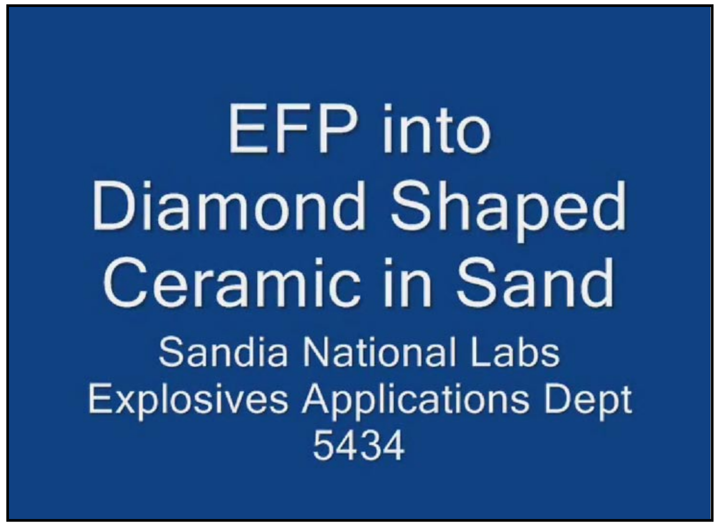
B.E. Bagwell, D.V. Wick, R. Batchko, J.D. Mansell, T. Martinez, S.R. Restaino, D.M. Payne, J. Harriman, S. Serati, G. Sharp, and J. Schwiegerling, "Liquid Crystal-Based Active Optics," in *Proceedings of the SPIE Optics and Photonics*, August 2006.

The Physics of Threat/Target Interaction for Advanced Armor Development

93605
W. V. Saul, G. A. Knorovsky, D. A. Hinkley, S. J. Glass, R. M. Brannon, V. S. Berg, L. R. Payne

Project Purpose

The primary purpose of this project was to draw upon the unique capabilities, facilities, and personnel available at Sandia to assist in mitigation of explosive threats being increasingly used against US protective forces and allies. Although a substantial amount of work is being done to design next-generation materials that will offer incremental improvements in armor function, optimized use of existing materials is an area where immediate benefit can be realized, and an area where Sandia is uniquely qualified to participate. Additionally, Sandia's role as a systems engineering laboratory allows a complete understanding of the evolutionary aspect of explosive devices, the intent being to minimize the reactionary method of armor development as it is currently performed.



This movie shows the effectiveness of a ceramic/sand matrix system (this particular layer is 1″ thick) in mitigation of EFP threat.

Forces face an evolving enemy that is acquiring weapons with capabilities beyond conventional blast and fragment behavior associated with improvised explosive devices (IEDs). Sophisticated devices are being employed including flyer plates, explosively formed projectiles, shaped charges, and long rod penetrators, each of which has secondary effects similar to IEDs. These device effects span a large range of penetration physics from conventional mechanics to hydrodynamic effects, and characterization of these various threat-target interactions is a fundamental requirement in the development of efficient, effective armor systems.

Timely development of armor systems is achievable through the unique combination of physics-based modeling, material science/engineering, and a comprehensive understanding of explosive device behavior available at Sandia. This project draws upon Sandia's unique capabilities in the areas of threat characterization and materials engineering through analytic and empirical studies. Our approach combines better characterization of current and potential battlefield threats (particularly penetration ability, standoff effectiveness, and collateral damage effects) with improved armor effectiveness through materials that disrupt impact-induced shock waves and deflect penetrating material.

To this end, we are investigating armor systems that include layers of dissimilar impedance materials and matrices of discontinuous fiber and ceramic compositions. Characterization of threat interactions with these developmental materials will optimize armor packages to better protect against multiple threats. This effort is intended to contribute to battlefield effectiveness by creating novel mitigation concepts and methods for current equipment and troop threats, while positioning Sandia to contribute in an anticipatory rather than reactionary mode to evolving threats.

FY 2006 Accomplishments

- Explosively formed projectiles (EFPs) are an increasingly used device in insurgency attacks, and no real systems for protection against these threats currently exist. We were surprised to learn from current experts that the area of EFP phenomena and understanding (industry wide) is quite limited. We began a limited scope EFP design and manufacturing effort that has yielded a repeatable, representative EFP design, and given us expertise in the area of EFP effects and optimization.

- The basic layered armor system we proposed has shown to be quite effective in initial simulation tasks. Relatively thin layers (1") achieve 50 percent reduction in velocity of an EFP. This is particularly noteworthy because even at these thicknesses, the system reduces residual velocity to a level where existing high-strength/low-weight materials can be employed to completely mitigate the threat. This proves that the concept of better usage of existing materials through proper positioning and layering will in fact be effective in improving armor performance.
- We initially overestimated the current capabilities of constitutive modeling in the areas of ceramics and crushable materials (sand). Several issues relating to model performance were identified. Many of these issues have since been rectified, which will allow subsequent progress on this project and other future simulation tasks with similar materials. Similar issues related to meshing and executing the code have been identified and are being rectified as necessary for threat/armor design activities.
- We have engaged contacts in the Department of Defense (DoD) and obtained feedback on armor systems, how they are being fielded, and so on, which will allow us to be more effective in optimizing both the results of this project and future efforts with DoD.

Significance

- Our efforts allow Sandia to successfully propose some emerging armor designs to the US Army Agile Equipment Design and Experimentation group for inclusion in FY 2007-funded activities.
- Several shortfalls in hydrocode simulation capabilities were identified, many of which have been rectified. This will benefit mission areas requiring multimaterial Eulerian/ Lagrangian interactions simulated in a hydrocode environment.
- For threat characterization early in the project, we needed to define and categorize the suite of high explosive threats to be considered for mitigation efforts. We chose the EFP as the primary design threat for several reasons; however, we quickly realized that the background in EFP design and function is severely limited. As a result, we began a limited scope EFP design and manufacturing effort that has yielded a repeatable, representative EFP design, which is currently being used in other research and development efforts. This has further positioned Sandia as an expert in the field of IED threat behavior and mitigation.

The original intent of the project to use existing materials to improve armor design has already proven promising and will likely benefit US forces in the near future.

Multiscale Behavioral Analyses of Integrated Surety Designs

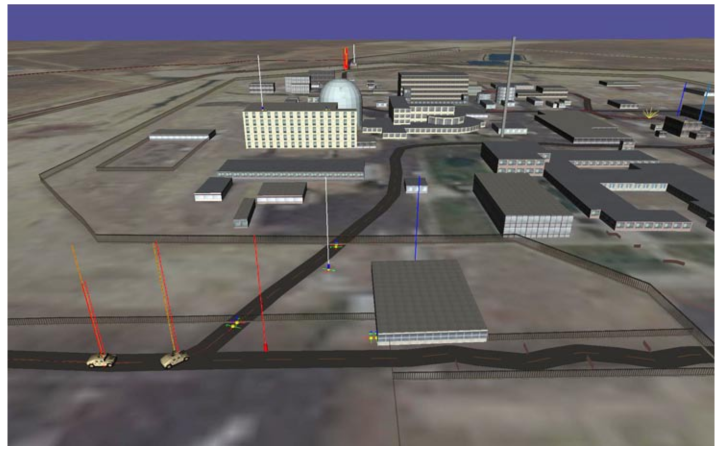
93637

S. Tucker, G. D. Whitford, M. J. De Spain, R. M. Griego, M. J. McDonald

Project Purpose

The purpose of this project is to develop a new systems analysis (SA) framework for the analysis and engineering of complex systems. It includes:

- Testing existing static and dynamic analysis tools against a complex systems analysis focus problem to build a better understanding of their strengths and weaknesses
- Creating new processes and methods for integrating the tools
- Testing the resultant processes
- Creating or specifying new tools needed to fill the gaps.



A DANTE run at a secure facility.

The goal is to improve the way Sandia develops high-quality answers that help decision makers make better system investment choices in addressing nuclear weapons integrated surety and improvised explosive device (IED) defeat. Sandia, industry and the Department of Defense (DoD) are analyzing problems of this nature with static analysis techniques ranging from informal (nodal) to detailed analyses (IDEF, a specialized modeling language and UML, or the Unified Modeling Language). They focus upon static structure analysis and hierarchical decomposition of requirements, composition, and networks. We found that the static techniques are useful but not well understood in complex system work, and are inappropriately matched to the dynamic, layered, multi-attribute, adaptive issues in systems being analyzed.

This project is determining the best way to use and combine UML and its successor SysML with dynamic simulation (Sandia's Umbra/DANTE) to drill down on system questions. This work builds upon key Sandia strengths and is being executed at both theoretical and practical levels.

Sandia has unique leadership in UML-based static analysis methods, technical depth in dynamic analysis (Umbra and DANTE), and ownership/must-solve focus of forcing problems (surety). We have chosen IED defeat as a focus problem as it has the right complexity and provides needed traction on IED and surety problems. This effort is the first to integrate and improve UML-based static analysis with dynamic analysis and apply them to IED defeat or integrated surety. The combined toolsets will have application beyond these problem focuses.

The main technical thrusts of this project are to:

- develop a SA methodology that supports both life cycle design and analytical processes, and addresses the separate issues of static and dynamic systems analysis
- identify and bridge missing systems analysis science elements
- integrate existing static and dynamic SA tools and technologies that support this form of complex systems analysis
- exercise the integrated tools on an integrated surety problem.

This year we worked to adapt and refine a static model of the IED problem writ large. We developed a UML model that identifies our adversaries' high-level goals and addresses specific threat operational concepts (CONOPS). We collected and began modeling coalition force issues including protective force and US Marine convoy security tactics, techniques, and procedures (TTPs), and technology options for intelligence, surveillance, and operational responses. Finally, we began extending our DANTE behavior engine and coding specific behaviors that we described in UML into the DANTE tool. Additionally, we began addressing the science aspects of analysis to support complex systems design.

FY 2006 Accomplishments

This project's first tasks were to 1) analyze initial mappings of static to dynamic analysis problem, 2) review SA literature, 3) develop, extend, and test baseline static to dynamic analysis strategy, and 4) develop static and dynamic models.

We adapted and refined a static model of the IED problem. A key issue in military, security, and security system analysis (including analyzing the IED problem) is in addressing vulnerabilities. Vulnerabilities derive from opposing force capabilities (i.e., the threat) in the context of opposing force objectives (i.e., the targets). In security design, the objective is to modify the target to be resistant to the threat. In other areas, the objective is to address the threat proactively. In both cases, it is imperative that the analysts understand the priorities, opportunities, and options that the opposing forces have in attacking our assets.

In this activity we developed a UML model from previously developed nodal and IDEF models. This part of the model identifies our adversaries' high-level goals. We then refined the model to address specific threat operational concepts. For example, we developed UML descriptions of the processes of organizing an ambush, attacking a convoy, and exfiltrating personnel and media products after attack. In addition to modeling the insurgents, we collected and began modeling (at a notional level) issues relating to our friendly forces. This includes protective force and US Marine convoy security TTPs. We also included behaviors relevant to the nuclear weapons generative modeling "GenMod" project.

Concurrently, we studied technology options for intelligence, surveillance, and operational responses. Finally, we began extending the GenMod behavior engine and coding specific behaviors that we described in UML into the GenMod tool.

Additionally, we began addressing the science aspects of analysis to support complex systems design. Our work has included consulting with Sandia design and analysis organizations, studying a wide breadth of emerging system analysis and engineering methods, developing a process for the analysis of system designs, and engaging key stakeholders and potential customers.

Significance

This project addresses defense missions in several ways: It develops a fundamental improvement to tool suites and methods for analyzing complex systems; provides a means of capturing decision makers' intentions and real-world constraints; and ensures that the resulting system design behaves as desired. The exemplar problem of IED and subsequent small arms attacks on a transport convoy is applicable to both DOE (Integrated Surety) and DoD (Iraq insurgency).

There is a growing realization that surety is not primarily a function of weapon features, but involves the entire environment surrounding the weapon as well as the weapon itself. Consequently, the understanding of the operation of surety features is becoming not just distributed, but also "graded," depending on the perceived threat condition. This entire approach requires totally new capabilities not only to evaluate existing systems and proposed enhancements, but to also understand the implications for developing totally new system wide solutions. Through this project we seek to provide a broad integration of system engineering and analysis tools that will generate a multiperspective understanding of the cost/benefit trades for surety in the future stockpile. The GenMod team has begun using our UML modeling process to systematically develop behaviors for their efforts.

An early result of this work has been the leverage of other Sandia tools in analysis activities that support complex systems design. One result is that we are developing concepts for an integrated modeling and simulation approach that spans from requirements through design to verification and testing of weapons systems. Umbra and ASSET (a Sandia-developed weapon controller design process) are being considered for early integration to support this effort.

Decision Framing and Characterization Approaches for Complex Security Environments

93638

J. T. Ringland, A. S. Yoshimura, N. J. Gleason, H. R. Ammerlahn

Project Purpose

Systems analysis has used a wide range of approaches to support an equally wide range of decision and resource allocation issues surrounding many types of national security issues. In planning this project, we initially divided those approaches into "analysis" – those based on simple but abstracted characterizations of a problem – and "simulation" – those based on more realistic, transparent representations of the dynamics of situations about which decisions are being made. The analysis and simulation efforts often operate quite separately from each other, with different teams, using different techniques, addressing different problems. While certainly the insights from one group is used by the other, real coupling and synergy is uncommon.

The purpose of this project is develop a richer characterization of system analysis model types and the functions they serve, so as to identify the conditions under which more linkage may and may not be reasonably expected, and then to propose and demonstrate, on a small sample problem, a process to implement a more linked analysis-simulation pathway.

FY 2006 Accomplishments

Based on a series of interviews with principals in a variety of large systems analysis modeling and simulation activities, some going back to the 1980s, we developed a three-axis characterization of model types:

- Abstraction <--> Representation (4 ordered categories)
- Project Focus <--> Capability Focus (3 ordered categories)
- Model Function (4 categories, without ordering)

plus a hierarchy of systems analysis functions, each of which was broken out into sub-functions:

- Concept Definition
- Concept Development and Design Support
- External Service Roles
- Program Development

From this, we were able to characterize where past activities fit and to more generally identify potential entry points and development paths. We noted a variety of interactions between the more abstract and more representative modeling activities, some of which were obvious (analysis can define what should be simulated), and some of which were less so (demonstrations, exercises, and training, even with inaccurate simulations, can elicit operational insights and open discussions in communities far removed from the traditional modeling community, which can in turn, enable a abstraction and analysis that was not possible before the simulation activity revealed issues).

We noted a variety of observations and issues with previous modeling efforts (technically and managerially), which helped us to define opportunities for improving modeling and simulation processes. The role of visualization and realistic depiction of analytical insights for communication with collaborators was noted as a particularly important area for focused design. Another key decision that drives the effectiveness of the modeling tools is the level of resolution chosen for the components of the systems model. Ongoing interaction and iteration between subject matter experts and tool developers, rather than a single-pass hand-off of requirements, was seen as highly valuable.

A process to address these issues was addressed in the prototyping phase of the project. In this final step of the research, we derived requirements and an initial communication module to address issues associated with response to the explosion of an improvised nuclear device in an urban area.

Significance

The characterization of system analysis-related modeling and simulation activities can help improve the processes underlying the decision support done by the systems analysis community, and can thus improve the quality and breadth of that support itself. This improvement manifests itself in several ways.

First, by identifying what types of modeling efforts fit with what types of problems, it may be possible to better or more quickly target the right tools and skilled individuals to a problem. It should be noted, however, that with the ill-defined nature of many systems analysis problems, it will never be possible to guarantee the first attack on a problem area will be the most successful one. Still, better characterization of the tools should speed that iteration.

Second, by following through on recommendations for improving modeling and simulation processes, the quality of those processes should be improved.

Finally, the entry and development path partially demonstrated here has the potential to grow into a new paradigm for linked modeling and simulation work. This can, in turn, expand the breadth of tools to be applied to systems analysis decision support.

Nonproliferation and Assessments

Monolithic Reconfigurable Radio Frequency Microelectromechanical (RF MEMS) Antennas

67088

L. M. Feldner, C. W. Dyck, C. T. Rodenbeck, B. H. Strassner II, C. D. Nordquist

Project Purpose

The purpose of this project is to explore the fundamental and technical limitations in electrically small and reconfigurable antennas. This work is important because a critical requirement of many miniature systems is the ability to sense and/or transmit electromagnetic energy for communications or remote sensing. This is accomplished using radio frequency (RF), microwave, or millimeterwave antennas, which can be fabricated with other electrical/mechanical components to yield a new class of reconfigurable antennas capable of multiband operation, adaptive beamforming, jamming/interference mitigation, polarization diversity, low probability of detection/interception communication, and direction of arrival estimation.

This project combines Sandia's growing expertise in RF microelectromechanical systems (MEMS) research and solid background in microwave radar, RF tag, and communication system design to develop revolutionary integrated antenna technologies that will enable next-generation applications. One aspect of this work is to demonstrate electrically small, tunable antenna prototypes at lower frequencies amenable for low-cost RF communication. Because these antennas can be tuned using commercially available solid-state components or packaged high-performance RF MEMS switches, the antenna design and demonstration can be performed with relatively low risk.

Another aspect of the project, supporting the previously mentioned antenna work, is to demonstrate RF MEMS switches in chip-scale hermetic packages suitable for integration into these circuit-board-based, centimeter-scale antennas. Additional investigation into low-voltage to high-voltage conversion circuitry is also essential for the integration of RF MEMS switches into common low-voltage electronics.

A final high-risk task on this project is to demonstrate a monolithic antenna array using RF MEMS loading capacitors to tune both the antenna element resonant frequency and RF MEMS phase shifters to steer the antenna beam. This work involves fabricating RF MEMS on two different substrates and wafer-bonding these substrates to a third wafer to realize a monolithic reconfigurable antenna capable of tuning from 30 to 40 GHz. The monolithic antenna approach offers the accuracy and small size associated with microfabrication for reproducible, repeatable, high-frequency antennas.

FY 2006 Accomplishments

We demonstrated an ultrahigh frequency (UHF), reconfigurable, electrically small antenna suitable for integration into standard circuit board materials. We developed this reconfigurable electrically small capacitively loaded planar inverted f-antenna (PIFA)-as-a-package (PIFA-AAP) to demonstrate the potential utility of reconfigurable antenna technologies to miniature and/or portable UHF wireless devices.

The scalable PIFA-AAP concept involves integrating the antenna and the device package to maximize the effective area of the antenna given the physical constraints of the application. We developed an approach to frequency-agility using commercial-off-the-shelf solid-state switches, overcoming the key weakness of extreme environmental sensitivity inherent to any electrically small antenna.

Measured performance includes near-contiguous tuning coverage between 407.8 MHz and 463.1 MHz with a total realized gain of better than -10 dBi across the tuning range. The measured noninstantaneous ensemble bandwidth of our proof-of-concept frequency-agile PIFA-AAP is superior to the Wheeler-Chu-Mclean fundamental limit for a single resonant antenna.

We also demonstrated RF MEMS switches in chip-scale hermetic packages suitable for integration into reconfigurable antennas and other circuit-board level reconfigurable circuits. The package is realized using a gold-tin preform to attach an alumina lid over the switch body and copper-tungsten filled through-substrate vias for the RF and bias signals. The packaged switch has < 0.5 dB insertion loss and better than 15 dB return loss through 10 GHz and occupies a volume of 2.6 mm x 2.5 mm x 0.75 mm.

To support the integration of high-voltage RF MEMS components into low-voltage systems, we demonstrated DC-DC converter circuits using only commercially available components on a simple circuit board. The circuits converted 3 V to 100 V while consuming approximately 20 mW of power. While the demonstration circuit is too large and consumes too much power for current applications, it demonstrates the current state of the art and provides a starting point for future efforts to decrease the voltage conversion circuit size and improve the efficiency.

We made progress toward, but did not complete, the monolithic antenna array. We experienced difficulties in the areas of through-wafer via holes in quartz substrates, silicon nitride switch encapsulation, and gold-tin wafer-level bonding that prevented the successful demonstration of a functioning monolithic antenna. While we did not demonstrate the final antenna array, the knowledge gained through this activity will be valuable for future RF and microwave projects requiring wafer-level bonding or packaging.

Significance

These results are significant in the context of advancing the state-of-the-art in electrically small and reconfigurable antennas, an area of interest in the commercial and military markets, and are substantial in the context of the current status of the technology. This work could have substantial impact to Sandia missions in the areas of communication, radar, tags, and any other system requiring RF antennas. The knowledge gained from this project may be applied to a variety of applications requiring miniature and reconfigurable antennas.

Other Communications

L.M. Feldner, C.T. Rodenbeck, and C.G. Christodoulou, "Tunable Electrically Small UHF PIFA-as-a-Package," in *Proceedings of the IEEE Antennas and Propagation Meeting*, p. 107.1, July 2006.

L.M. Feldner, "Reconfigurable RF MEMS Antenna Arrays and Electrically Small Antennas," University of New Mexico PhD Thesis, Albuquerque, NM, November 2006.

Risk Assessment Meta Tool

67090
A. M. Bouchard, W. D. Henry, G. C. Osbourn

Project Purpose

The purpose of this project is to create a risk assessment metatool that imports and consolidates data from multiple risk assessment tools that don't "talk to each other" into a single environment and then allows the user to generate new analyses on the fly.

The first version will allow the user to import multiple files from the Joint Conflict and Tactical Simulation (JCATS) and construct on-the-fly analyses from these files. In the second version, we will add the capability to import files from the Adversary Time-Line Analysis System (ATLAS) and develop analyses that combine data from JCATS and ATLAS.

In addition, we will provide the user the ability to specify a file format so that the code to import a completely new file type can be self-assembled on the fly. The end result will be a metatool with which the user can import files from multiple risk assessment tools and create analyses that combine data from these various tools rapidly in response to changing needs.

FY 2006 Accomplishments

We designed and implemented the autogeneration and self-assembly of executable code. The self-assembling software (SAS) generator not only generates and executes standard commands like math functions, searching, and so on, but can also self-assemble complex sequential or even hierarchical commands (subroutines). The SAS generator took 11 man-months to design and implement, resulting in over 11,000 lines of code (in addition to 11,000 lines for the self-assembling building blocks themselves). Test examples of autogenerated code were real JCATS analyses performed by JCATS analysts.

We designed and implemented an auto-save/auto-load capability. This capability automatically saves new data, objects, and results to the hard disk, eliminating the need for the user to do so. It also loads all the data necessary to render and interact with interface objects on demand. Together, these capabilities enable the user to interact only with the entities on the interface (books, bookcase, and so on) and not worry about names or locations of their data on the hard disk.

Additionally, we refined the design of the graphical user interface for programming in English-like sentences. Implementation of the prototype metatool is still under way.

Significance

As we near the completion of a prototype risk assessment metatool, we are preparing to test the software for usability. Our development has been aimed at security analysts and others who need to rapidly develop custom software in response to changing threats or problems. Our accomplishments bring us much closer to being able to address this unmet need of vulnerability and risk analysts, whether their problem domain is nuclear facilities, other infrastructure assets such as power facilities or the US Mint, intelligence information, or other problems of national security.

Vulnerability Assessment with Dynamic Reverse Engineering of Embedded Processors through Innate Debug Mechanisms of System-on-Chip Integrated Circuits

67096

J. J. Clement, J. V. Wolf, A. Phan, J. M. Green, E. I. Cole Jr., J. L. Etzkin

Project Purpose

The use of system on chip (SOC) integrated circuits (ICs) is increasing in embedded systems. This trend is having a severe negative impact on our capability to perform effective vulnerability analyses of these systems. SOC ICs contain multiple embedded cores, including processors, read-only memory, and random access memory. Vulnerability analysis on embedded systems using SOCs is prohibitively expensive to perform. A promising solution lies in the manipulation of the boundary-scan interface, which was originally created to test interconnections between ICs on a printed circuit board.

An IEEE standard for boundary-scan testing has been specified by the Joint Test Action Group (JTAG). By adding to the five commands required by the standard, the IC designer can enhance boundary-scan testing with emulation and in-system programming. This greatly enhances the designer's ability to create software tools to check circuit functionality and debug. Unfortunately, access to these internal development tools and the supporting documentation is often not available to external users.

The purpose of this project is to discover novel techniques to quickly identify the JTAG interface pins to allow us to perform focused reverse engineering of SOCs by leveraging our current analysis capabilities. Early on we found that it was impractical to use physical deprocessing to accomplish these objectives. This capability does not exist at Sandia, and it would have been prohibitively expensive to develop in this project. Our focus then shifted to less destructive methods, based on electrical testing.

With the pins identified and JTAG enabled, we plan to use available commercial off-the-shelf tools developed for the individual cores. Emulation will give us the dynamic capability to obtain a software trace, and thus to focus on critical areas.

FY 2006 Accomplishments

We generated a nondestructive three-step procedure to identify the JTAG interface pins on an unknown SOC IC. When the JTAG pins are known, we use a JTAG state machine analyzer we constructed that parses and analyzes captured JTAG traffic and converts it into readable JTAG instructions. Finally, we developed hardware and software to provide a tool that can be used to communicate with and control a target device through the JTAG interface.

Significance

We studied how several IC manufacturers implement the JTAG interface on a variety of devices. This knowledge will provide a strong basis for future IC vulnerability assessment projects. The hardware and software that make up the JTAG controller tool we developed can be easily adapted and used in future projects and has already proved useful in a couple of current projects.

We believe that the techniques and procedures we formulated to identify JTAG lines on an unknown IC will find application in the very near future and provide us with a nondestructive alternative to perform vulnerability assessments on SOC ICs.

Nonlinear Optical Detection of Biological and Chemical Aerosol Agents Using Femtosecond Lasers

67098

J. Urayama, M. W. Kimmel, N. D. Zameroski, J. Urayama, M. L. Naudeau, E. A. Disch

Project Purpose

Spectroscopy using ultrashort pulse lasers is a promising technique to remotely obtain information about physical parameters in bioaerosol or chemical plumes, including estimation of size distribution, refractive index, and composition spectroscopy. The unique combination of wavelength diversity, coherent bandwidth, and impulsive coupling – attributes characteristic of femtosecond laser technology – provides the potential for capable and robust next-generation stand-off detection in aerosol and topographic measurement environments using new signature paradigms employing both temporal and spectral discrimination.

Active broadband imaging technology will enhance scene contrast by noncooperative conversion to reflectance independent of ambient light conditions and will allow estimation of aerosol size distribution properties using multispectral elastic backscatter and wavelength-dependent extinction in the laser radar inverse equation. Adaptive pulse shaping of the bandwidth can be used to customize molecular excitation conditions for a broad range of applications including quantum coherent control of molecular vibrations, continuum generation for multitrace gas analysis, and random frequency hopping to avoid detection by threat warning systems.

Propagation control strategies employing short-pulse lasers can be used to controllably generate intensity at range to initiate nonlinear spectroscopic processes. By exploiting optical nonlinear effects such as multiphoton-excited fluorescence (MPEF), stimulated Raman scattering, and laser-induced plasma breakdown in aerosol droplets it is possible to enhance backscatter signatures for improved collection and achieve spectral specificity in chromophores using near-infrared lasers with reduced atmospheric transmission losses. The internal focusing properties and spatially resonant microcavity modes with an individual aerosol droplet excited by a femtosecond pulse will further enhance nonlinear optical conversion and reduce corresponding thresholds for signal generation in the power-dependent cross section.

This project describes proof-of-principle laboratory experiments to evaluate feasibility of remote sensing using ultrashort-pulse lasers. Calibrated experiments of surrogate systems will be used to baseline performance models and assess operational capability. In FY 2006, we concentrated on the exploration of nonlinear effects on surrogate spore materials and investigations in broadband linear remote sensing techniques using femtosecond lasers.

FY 2006 Accomplishments

We conducted laboratory experiments calibrated to identify and measure candidate nonlinear aerosol signatures in well-characterized spore surrogates using MPEF, and linear-induced fluorescence exploiting third harmonic generation in air.

In order to address the study of the biological surrogates, a laboratory at Sandia was certified for working with BSL-1 (biosafety level 1) biohazards. Samples examined included *Bacillus globigii* and *Bacillus megaterium*, which are surrogates for *Bacillus anthracis* (anthrax). Initially, we performed experiments in both spore solutions and in raw tryptophan and tyrosine solutions. Experiments yielded nonlinear fluorescence from the raw material solutions; however, the spores yielded only linear fluorescence.

This raised significant questions regarding the impact of spore morphology on the generation of nonlinear signatures. To this end, we constructed an electrodynamic trap in order to isolate individual spores for further studies. We anticipate that this apparatus will be used in further studies.

We performed similar studies on chemical surrogates, including thiodiglycol and pinacolyl methylphosphonic acid (surrogates for mustard gas and soman, respectively). We observed and recorded stimulated Raman signatures for both materials. However, we also recorded MPEF signatures for both surrogates, which were not expected in these materials.

The signatures varied from manufacturer to manufacturer and are, therefore, believed to be processing-dependent, potentially due to a contaminant in the manufacturing process. Although these represent potentially new signatures, they offer no specificity with regard to unique identification of the chemical agent.

We performed specificity studies using the coherent anti-Stokes Raman spectroscopy (CARS) technique. We performed calibration measurements in benzene, measuring photon yields and angular dependences, and additional measurements in dipicolinic acid (DPA), which is present in anthrax spores.

DPA exhibited a unique CARS signature in the temporal domain, which indicates potential for species identification from standoff. However, resolution of this signature requires a large dynamic range on the measurement, which raised questions regarding the potential to measure this from standoff. Furthermore, signatures had a strong dependence on solvent material, which led to important questions regarding target morphology (spore, liquid, and so on).

We constructed an ultrawide bandwidth source consisting of a femtosecond oscillator and a photonic crystal fiber. We conducted experiments to examine the source characteristics and propagation performance of this source, in order to assess the feasibility of its application. Preliminary laboratory measurements up to 10 m, using simulated atmosphere, demonstrated minimal impact on source characteristics. Integration times shorter than 1 ms demonstrated no impact of the simulated atmosphere. Longer integration times are achievable only when active source stabilization is employed. This indicates that one of the main constraints related to the applicability of broadband light is source driven, not propagation driven.

Significance

Ultrashort-pulse lasers offer a great potential to revolutionize remote sensing applications. Nonlinear signatures provide a means for out-of-band excitation that allows for covert measurements. CARS offers potential for unique identification of a given material, which when used in combination with femtosecond lasers and adaptive pulse shaping can provide a means to signature isolation against a cluttered spectral background. This would lead to not only a new signature set, but also the means to perform measurements in previously inaccessible scenarios, which could expand US capabilities tremendously.

At the completion of this project, questions remain regarding target morphology and the practicality of standoff measurement, but as a result of this project, work for others (WFO) customers are funding further work in this area.

The use of wide coherent bandwidth for linear applications offers great potential for a variety of applications, not only for chemical/biological detection (e.g., broadband differential absorption light detection or DIAL), but also for a variety of intelligence surveillance and reconnaissance applications allowing expanded capabilities to enhance mission performance. The work on ultrabroadband sources and propagation performed under this project has spurned a great deal of interest from potential WFO and intelligence customers, and may lead to external investment in Sandia in the future.

Other Communications

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Compensation of Ionospheric Errors for Geolocation

79843

J. J. Mason, D. D. Cox, M. F. Prins, R. M. Holman, M. J. Navarro, C. L. Leger, L. Romero, R. M. Axline, C. R. Collins, D. D. Kiffer, R. Connell, A. Romero

Project Purpose

The purpose of this project was to advance the state of the art in compensation of ionosphere-induced errors that limit frequency of arrival-based satellite-borne geolocation systems.

FY 2006 Accomplishments

In FY 2005, we:

- set up a wide area augmentation system (WAAS) receiver
- established a WAAS database
- developed a procedure to smooth and differentiate WAAS total electron content (TEC) data to produce real-time estimates of TEC rate
- stood up a ground station to establish a radio frequency (RF) test link with several satellites to conduct tests of the compensations.

We used burst detector analysis-equipped global positioning system platforms configured to transpond ultrahigh frequency signals. We assembled a ground station with three available 14-foot reflectors. We transmitted a carrier through this earth-space-earth communications channel and measured the difference in the received carrier frequency from the expected frequency.

This year we improved our frequency measurement accuracy to less than 1/10 of a Hz so that we could measure any significant ionosphere doppler. However, we never observed any ionosphere dopplers large enough to be measured. We documented our detailed characterization of the RF links in a SAND report.

Significance

We established how accurately the frequency of a carrier in our RF channel can be measured. This is equivalent to determining how accurately the position of a persistent narrowband RF transmitter could be determined.

Single-Photon-Sensitive Imaging Detector Arrays

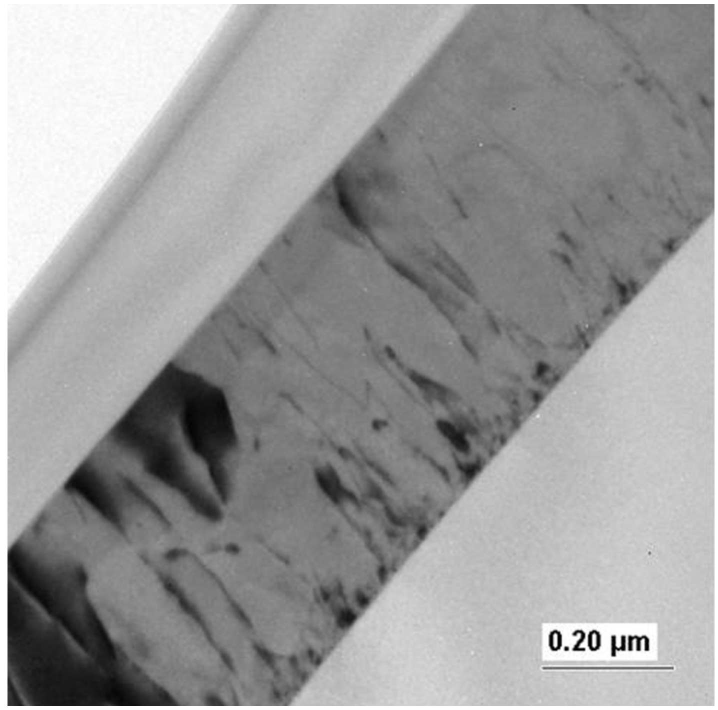
79844
M. S. Carroll, K. D. Childs, D. K. Serkland, J. F. Klem, K. M. Geib, I. J. Fritz, J. L. Rienstra

Project Purpose

The purpose of this project is to address a need for a short-wave infrared light detector for ranging (lidar) imaging at temperatures greater than 100 K. We fabricated several novel device structures to improve avalanche photodiodes (APDs) to achieve the desired performance at 1550 nm. A primary challenge to achieving high sensitivity APDs at 1550 nm is that the small band-gap materials (e.g., InGaAs or Ge) used to detect near-infrared (NIR) exhibit higher dark counts and higher multiplication noise than do other materials like silicon.

To overcome these historical problems, we designed and fabricated APDs using separate absorption and multiplication (SAM) regions. The absorption regions used InGaAs or Ge to exploit the sensitivity of these materials at 1550 nm. We chose Geiger mode (GM) detection to circumvent gain noise issues in the III-V and Ge multiplication regions, and built a novel Ge/Si device to examine the utility of transferring photoelectrons in a silicon multiplication region. Silicon is known to have excellent analog and GM multiplication properties. The proposed devices represented a high risk for a high-reward approach. Therefore, one primary goal of this work was to experimentally resolve uncertainty about the novel APD structures.

This work specifically examined three different designs: 1) We proposed an InGaAs/InAlAs GM structure for the superior multiplication properties of the InAlAs. The hypothesis to be tested in this structure was whether InAlAs provide an advantage in GM; 2) We proposed a Ge/Si SAM representing the best possible multiplication material (i.e., silicon); however, significant uncertainty existed about both the Ge material quality and the ability to transfer photoelectrons across the Ge/Si interface; and 3) We proposed a third pure germanium GM structure because bulk germanium has been reported to have better dark count properties. However, significant uncertainty existed about the quantum efficiency at 1550 nm.



Ge-on-Si epitaxy grown by plasma enhanced growth

FY 2006 Accomplishments

This work specifically examined three different designs of APDs that showed promise of meeting the required single photon sensitivity desired for nonproliferation applications. Because these designs represented innovative ideas to overcome existing technical challenges, they also represented risk due to uncertainties in untested aspects of each structure. To test the structures, fabrication and measurement of devices was necessary.

In FY 2005, we developed new fabrication processes to build the Ge and Ge/Si devices, fabricated first generation InGaAs/InAlAs APDs, and initiated measurements. In FY 2006, we completed and measured the Ge and Ge/Si devices, and gained a better understanding of the capability in GM measurement.

We demonstrated a Ge/Si separate absorption and multiplication region that has very high gain in the silicon and absorbs 1310 and 1550 nm light in the Ge.

We further accomplished transfer of NIR photoexcited electrons into a silicon multiplication region with relatively low dark current despite having high defect densities in the epitaxial germanium.

We further demonstrated GM operation of the Ge/Si device, which indicated that the device can be sensitive to single photon excitations. However, these first-generation Ge/Si devices have low internal quantum efficiency, and the dark currents do not scale ideally with temperature, making it difficult to achieve the desired noise performance.

We attribute the nonideal dark current to tunneling through oxide defects observed at the Ge/Si interface, and attribute the low quantum efficiency to higher recombination in the defective Ge than predicted, which could be due to slow electron mobilities produced by unexpectedly high defect trapping. Both tunneling and recombination can be significantly improved; however, the exact magnitude of improvement is unknown, leaving remaining uncertainty about whether this structure can achieve the desired performance even with further development.

GM detection in InGaAs/InAlAs, Ge/Si, Si, and pure Ge devices fabricated at Sandia was shown to overcome gain noise challenges of narrow bandgap APD materials. This included the completion of a fully operational single photon GM measurement setup. This represents important learning at Sandia that will enable us to respond to future single photon detection needs for imaging, communications, and computing for nonproliferation applications.

Significance

No single semiconductor material has the necessary combination of properties (e.g., bandgap, intrinsic carrier concentration, and ionization coefficients) to produce an APD that has a quantum efficiency better than 10 percent at 1600 nm, low noise, and high enough gain to satisfy the future demands of some nonproliferation imaging applications. An established approach in APD design to circumvent the limitations of a single material is to use separate absorption and multiplication (SAM) regions. However, the combination of nonideal carrier ionization properties in III-V materials and challenges to lattice matching Si with a narrow bandgap material has prevented the demonstration of an ideal SAM-APD with both 1600 nm sensitivity and multiplication performance similar to silicon APDs. In order to overcome this challenge, we examined engineering a III-V material to improve multiplication properties and we pursued a novel technique to integrate a narrow bandgap material with silicon.

The further improvement of NIR single photon detection is also of interest to intelligence community areas like quantum key distribution (QKD) and quantum computing. APDs operated in GM are of particular interest for QKD networks because of their potential for more cost-effective scale-up compared to current proof-of-concept systems that use liquid helium cryogenically cooled superconductor-based detection schemes.

Current physics-level experiments use these systems, but they are impractical for implementation. The development of InGaAs/InP GM detectors has been the focus of the noncryogenic work. Germanium has not received as much attention despite its better after-pulsing behavior because historically the focus has been on 1550 nm detection for QKD. However, a recent desire for 1310 nm for QKD has made germanium and novel germanium heterostructures an appealing alternative for increasing quantum-bit rates.

Development of germanium integration on to silicon is also of great interest for a growing set of applications, including 1) silicon photonics, 2) low-cost, high-density NIR imaging, and 3) solar cell applications. The integration of germanium detectors and modulators with silicon waveguide technologies offers the potential to significantly impact areas such as navigation (optical gyroscopes), computation (analog computing and inter- and intrachip communication), and detection (low-cost, high-density NIR arrays). Furthermore, the development of low-cost and light-weight compliant Ge on Si substrates (to replace pure Ge substrates) for GaAs-based high-efficiency solar cells is of great interest to both satellite and terrestrial applications.

The development of these technologies, therefore, addresses a number of relevant and immediate national security issues. Funding has been obtained for a postdoctoral appointee to conduct a two-year follow-on project to improve NIR single photon detection. Additionally, as a result of this work, collaborations both with universities and companies active in this area were established.

Refereed Communications

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Y.S. Suh, M.S. Carroll, R.A. Levy, G. Bisognin, D. De Salvador, and M.A. Sahiner, "Implantation and Activation of High Concentrations of Boron and Phosphorus in Germanium," in *Proceedings of the Materials Research Society Symposium*, vol. 891, pp. 303-308, 2006.

M.S. Carroll, J.J. Sheng, and J. Verley, "Low-Temperature Heteroepitaxial Growth of Ge on Si by High-Density Plasma Chemical Vapor Deposition," in *Proceedings of the Materials Research Society Symposium*, pp. I09-02, 2006.

Y.S. Suh, M.S. Carroll, R. Levy, G. Bisognin, D. De Salvador, M.A. Sahiner, and C.A. King, "Implantation and Activation of High Concentrations of Boron in Germanium," *IEEE Transactions on Electron Devices*, vol. 52, pp. 2416-2421, November 2005.

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M. Carroll and R. Koudelka, "Accurate Modeling of Average Phosphorus Diffusivities in Germanium after Long Thermal Anneals," presented at the International SiGe Technology and Device Meeting, Princeton, NJ, May 2006.

M. Carroll and R. Koudelka, "Accurate Modeling of Average Phosphorus Diffusivities in Germanium after Long Thermal Anneals: Evidence of Implant Damage Enhanced Diffusivities," to be published in *Semiconductor Science and Technology*.

Fully Integrated Microfluidic Microthruster System for Micropropulsion Applications

79846

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Project Purpose

Miniaturization is becoming prevalent in the space satellite community to reduce mission cost and risk and create new roles for satellites. The goal of miniaturization is to produce small, inexpensive, and mass-produced systems without sacrificing the capability to perform highly complex functions. For instance, a constellation of nanosatellites, which are defined as satellites weighing 10 kg or less and about the size of a basketball, can operate as a wide-area sensor for earth-staring surveillance, or a single, highly maneuverable nanosatellite can serve as a repair, resupply, or inspection craft for other space vehicles.

For such missions to be possible, an efficient propulsion system is required. Nanosatellite system constraints on mass, volume, power, and fuel pose several challenges to designing an efficient propulsion system for orbit control and high-altitude maneuvers. The scale of macrocomponents in a conventional satellite propulsion system makes this technology unsuitable for nanosatellites, thus requiring new micropropulsion technology.

Recently developed electrokinetic (EK) pumps and microfluidic substrates for extreme pressure and temperature conditions may prove to be an enabling technology for microthrusters. We proposed a strategy to build a micropropulsion system using an innovative, but simple, architecture scheme suitable for the harsh conditions of space. The design uses EK pumps to deliver propellants for thrust generation because EK pumps have no moving parts and can be voltage-controlled for direct valveless metering of propellants. In our design, we coupled the pumps to a high-temperature, high-strength ceramic microfluidic platform with an integrated thrust chamber and exhaust nozzle to realize a small, lightweight micro-thruster package.

FY 2006 Accomplishments

- We tested the thruster components and developed a more fully engineered thruster prototype. Our achievements showed that EK pump micro-thrusters are a viable technology for miniaturized satellite propulsion.
- We fully characterized a silica EK pump for hydrazine rocket fuel, demonstrating that hydrazine can be electrokinetically pumped with good efficiency.
- We investigated alternative surface chemistry and surface coating for pumping hydrogen peroxide.
- We fabricated and tested capillary EK pumps and large format sintered monolith pumps in both monopropellant and bipropellant configurations.

We achieved direct pumping of hydrazine for efficient thrust generation in a monopropellant configuration by pressurizing the fuel to high pressure and releasing it through a cryogenic valve. We measured reproducible thrust values as high as 1 millinewton using a microbalance thrust measurment test stand.

As an alternative method, we used high flow-rate sintered monolith pumps to demonstrate controlled propellant delivery for thrust generation. Specific impulse (a measure of thrust to mass flow-rate efficiency) was as high as 120 sec for this prototype device.

We fabricated and tested low-temperature cofired ceramic microthrusters with integrated iridium and silver catalyst reactors. We used glass microfabricated devices using maskless lithography and direct-write femtosecond laser machining techniques to test geometries and alternative catalyst chamber designs.

Significance

This project represents a potential cross-fertilization between the nascent microfluidics and biotechnology programs focused on homeland security and traditional core missions in nonproliferation and assessment. The development of EK microthrusters would help to enable revolutionary persistent surveillance capabilities using nanosatellite clusters or constellations, widen Sandia's foundation in satellite technology, and complement other space microsystem programs. The development of advanced satellite technology capabilities is a key element in Sandia's mission to provide enabling technologies for the US intelligence community.

Refereed Communications

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Other Communications

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Standoff Detection of Explosives Using UV Lidar Technology

79847

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Project Purpose

As the use of explosives by terrorists continues and in some cases increases throughout the world, there is ever-growing interest in technologies that can detect explosives. Among the critical threats associated with the use of explosives, perhaps none is of greater concern than the large vehicle bomb (LVB).

LVBs are especially devastating because of the large masses of explosives material that can be contained in them – hundreds, thousands, or even tens of thousands of pounds, depending on the size of the vehicle. This compares to masses of a few tens of pounds that can be carried by a person such as a suicide bomber. While there are many scenarios involving the detection of explosives in vehicles, detection at a distance – so-called standoff detection – is of particular interest, because it allows personnel screening the vehicle to do so from a safe distance.

The purpose of this project was to investigate ultraviolet (UV) fluorescence lidar (light detection and ranging) as a technique for the standoff detection of explosives. In particular, it was envisioned that UV fluorescence lidar might be used as a technique to identify vehicles that contain LVBs by detecting adsorbed particle contamination on the exterior surfaces of a vehicle.

Previous studies regarding the loading of explosives into vehicles have shown that the loading process typically leaves detectable amounts of explosive contamination on the exterior of the vehicle in the form of fingerprints that contain particles of the explosive in question. The use of laser-based optical techniques such as UV lidar represents one potential means of detecting this trace contamination at standoff distance.

FY 2006 Accomplishments

In FY 2005 we investigated fluorescence from a number of explosive compounds and related materials commonly used in LVBs and other improvised explosive devices, including ammonium nitrate (AN), fuel oil (FO), ammonium nitrate/fuel oil mixtures (ANFO), 2,4,6-trinitrotoluene (TNT), cyclonite (RDX), and pentaerythritol tetranitrate (PETN). The primary findings were that TNT, RDX, and PETN show little if any native fluorescence but do exhibit a fluorescence quenching effect on some surfaces, while ANFO exhibits some fluorescence that originates not from the AN itself but either from the fuel oil or from additives/impurities in the oil or AN.

In addition, we investigated ANFO and its constituents using fluorescence microscopy to evaluate the possibility of particle and crystal effects on the fluorescence. The results of these studies were completely consistent with the laser-based studies. All of the fluorescence signals we detected could be attributed to the FO, the hygroscopic coating on the AN prills, or to trace metal impurities in the bulk AN.

The work performed in FY 2006 extended the research done in FY 2005, with an emphasis on AN, FO, and ANFO. We investigated the use of polarization of both the input excitation light and the fluorescence emission as a means to improve discrimination between the laser-induced fluorescence (LIF) from the explosives of interest and the LIF from the surfaces on which the explosive material was adsorbed. While we observed polarization effects on some substrates, the effects did not prove to be consistent enough to provide the basis for a robust, real-world detection technique.

Another area we investigated was the use of LIF imaging to determine whether spatial variations in the LIF from a surface could be used as an indicator of explosive contamination. Since the LIF from surfaces of interest – particularly vehicle bodies – tends to be quite bright, this amounts to looking for a quenching effect that results from the presence of explosives.

We had limited success, but the results were inconsistent, and quenching due to adsorbed explosives is probably impossible to distinguish from quenching or simple light-blocking due to other surface contaminants such as dirt. Therefore, we do not consider fluorescence quenching by explosives to be an exploitable signature for standoff detection.

We considered, but decided not to pursue, studies of the fluorescence of triacetonetriperoxide (TTAP) after a literature search uncovered several previous studies that found this molecule to exhibit no fluorescence.

Significance

Based on our results, it appears unlikely that UV lidar represents a robust, real-world technique for the standoff detection of LVBs. None of the explosives we investigated exhibited fluorescence signatures that are strong and unique enough to provide reliable, specific “fingerprints” for the species in question.

The nitrate-based explosives exhibit fluorescence quenching effects when adsorbed on some surfaces, but there are too many possible causes of a decrease in surface fluorescence for this to serve as a reliable indicator of the presence of a particular explosive. The various ANFO formulations we investigated show some fluorescence due to fuel oil, additives, and impurities, but none are consistent enough or sufficiently distinguishable from background to allow a determination of the presence of ANFO.

We do believe that the work performed represents the most detailed and careful study to date of the fluorescence and fluorescence quenching properties of TNT, RDX, PETN, and ANFO. Results on this topic in the literature are sparse at best, and our results are thus of considerable interest to those who study explosives detection and to the wider scientific community. We therefore believe that the final SAND report will be a valuable document. There is also a possibility that we will subsequently publish some of our results in a peer-reviewed scientific journal.

Fail-Safe Infectious Substance Transport Packages

79848
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Project Purpose

The objective of this project is to develop a safe and secure packaging method for the transport of infectious substances (IS) that will increase the likelihood that critical material can be shipped to appropriate laboratories following a bioterrorism event or the outbreak of an infectious disease.

Many carriers, concerned about leaking packages, refuse to ship IS. Air carriers are especially concerned, since leakage of IS may require decontamination of their aircraft – a difficult and costly task. Current packaging for shipping IS does not have the ability to neutralize or kill leaking IS.

The shipment of IS is carefully regulated, requiring a triple packaging system. The IS is placed in a securely closed, watertight, leak-proof, primary container labeled with the contents. Absorbent material is included immediately outside the primary container to completely absorb the contents in case of a spill. The primary container is then placed in a durable, watertight container that acts as a secondary container. The combined primary and secondary containers are then placed in an outer shipping container that protects the contents from physical damage and water during transport.

The packaging method we developed contains a novel decontaminating material that kills or neutralizes any infectious organisms that leak from the primary container. This self-decontaminating feature will alleviate concerns of both ground-based and air-based carriers and allow lower-risk shipment of IS.

Our method focuses on replacing the absorbent material in the current triple packaging system with a material that both absorbs and neutralizes IS should a leak occur in the primary container. The absorbent/neutralizing material is based on the fundamental chemistry of the Sandia decontamination foam (DF-200) that rapidly neutralizes chemical and biological warfare agents with a material that exhibits very low toxicity and corrosivity.

For this project, we are developing a material that uses Sandia's decontamination technology in granulated (powdered) form in combination with highly sorptive material to absorb blood or other infectious materials leaked from a container.

FY 2006 Accomplishments

We focused on incorporating the absorbent/neutralizing formulation into appropriate packaging. The current triple packaging system contains a watertight secondary container to contain a spill from the primary container (the primary container is a glass or plastic receptacle that contains the sample with pathogenic organisms). The objective of this task was to develop a method to pack the absorbent/neutralizing formulation in the space between the primary and secondary container or outside of the secondary container so that it will contain and neutralize leaks from the primary container. In current packages, this space is occupied by absorbent material alone.

We evaluated three approaches:

- Incorporate the absorbent/neutralization formulation into a pad or pouch configuration that can be wrapped around the primary container
- Configure the absorbent/neutralization formulation into tablets or beads that can be poured into the empty space between the primary and secondary container or outside of the secondary container
- Impregnate the absorbent/neutralization formulation onto tissue or other types of material that can be placed between the primary and secondary container.

The preferred approach was to encapsulate the absorbent/neutralization material into small beads and to pack the beads around the outside of the secondary container in a vented vessel. We tested this approach for efficacy against microorganisms, sorption capacity, and stability.

Significance

The DOE national security mission includes concerns of weapons of mass destruction, nonproliferation, and regional stability. Outbreaks of infectious diseases, whether natural or intentional, can impact regional stability. The international response to disease outbreaks requires safe and secure transport of infectious substances to identify samples, provide evidence for attribution, and support decisions to contain the outbreak.

Recent natural outbreaks of highly infectious diseases have had devastating consequences for public and agricultural health, the international economy, and international security. The outbreak of severe acute respiratory syndrome (SARS) in Asia in 2003, which infected more than 8,000 people and killed almost 800, ravaged economies in the Pacific Rim and Canada and struck fear across the globe. The outbreak of Foot and Mouth Disease (FMD) in the United Kingdom in 2001 caused economic losses of approximately $8 billion. Outbreaks of a zoonotic form of avian influenza in 2004 have also inflicted enormous losses on many Asian countries; recovery from these outbreaks will cost approximately $500 million.

The spread of infectious disease is not stopped by political boundaries and has the potential to destabilize regions. Regional stability would be greatly enhanced by packaging that facilitates rapid transport and identification of IS. Timely identification will help regional decision makers determine if they must respond to intentional dissemination of a biological weapon or if they are responding to a natural disease outbreak.

Although the transport of IS is vital to public health and national security communities, IS packages occupy a small percentage of the market for the transport of hazardous materials. The number of potentially truly dangerous pathogens being shipped is significantly smaller still. Hence, there is little market incentive for researching fail-safe package designs.

Our fail-safe packaging technology will enhance the ability to rapidly transport infectious substances, increasing the ability of the global community and regions of concern to diagnose, respond, and control outbreaks.

Development of Entomologic Surveillance to Aid Early Disease Detection (EDD)

79849

S. A. Caskey, H. A. Smartt, M. P. Wilson, M. Aspelin

Project Purpose

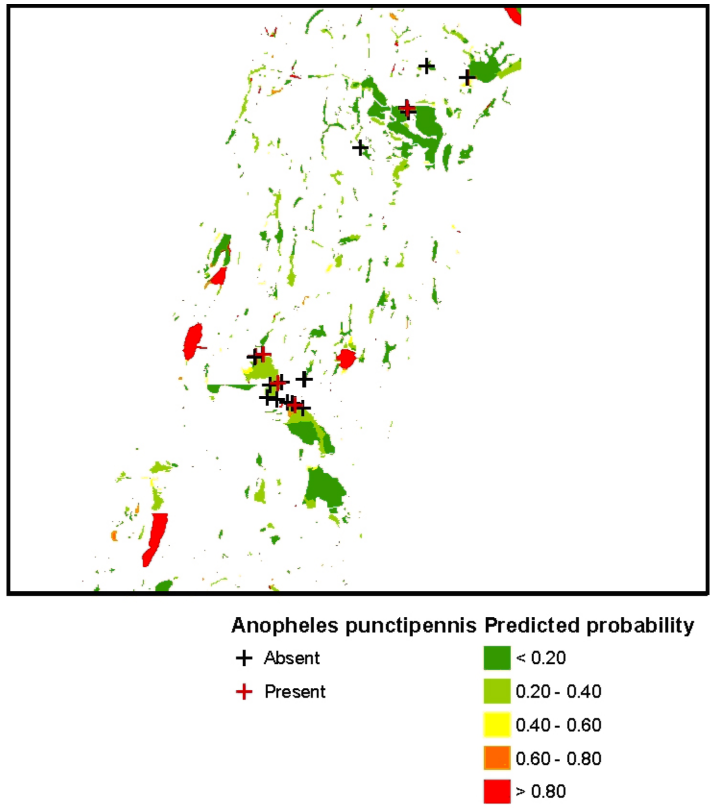
The purpose of this project was to review the potential use of remote sensing models to identify the likely habitat of certain insects, which could enable the early testing for disease and allow for timely and accurate mitigations measures. The ultimate goal was to develop a remote sensing model using a combination of imagery and insect data to aid in the detection and mitigation of vector-borne disease.

Many biological agents with a history of biological weapons use can be vector-borne. In this scenario, infected insects are released with the intention of causing an outbreak. In addition, approximately 17 percent of the global burden of infectious diseases is vector-borne. About 550 of these are arthropod-borne viruses (arboviruses), of which about 100 cause disease in humans.

For this project, we focused on vectors of West Nile virus and Eastern equine encephalitis (EEE): *Culex pipiens*, *Culex restuans*, *Culex salinarius*, *Anopheles punctipennis*, *Aedes vexans*, and *Culiseta melanura* within the area of New Haven, Connecticut. This area was selected because it is the location of our collaborating institution, Yale University, which facilitated groundwork, and because there is an abundance of mosquitoes in this part of North America

We focused on three major research areas:

- Identify a correlation between mosquito species and the habitat. While the general habitat is known for most species, the specific habit characteristics are not really established.
- Review remote sensing data from multispectral and hyperspectral images to determine which would offer the better information regarding a wetland habitat.
- Use data from the hyperspectral sensor, Hyperion, to determine if a model could be created that would identify a wetland habitat specific to a species of mosquito.



Predicted positive or negative wetlands for An. punct *larvae based on veg indicies from remote sensing data.*

FY 2006 Accomplishments

We created a rough species-specific habitat classification for the mosquito species *An. punctipennis* using collected larval samples as well as other ground data by Yale University.

The collection of larval samples within a two-mile buffer, identified by Connecticut Agricultural Experiment Station light traps, provided data including the species collected, the counts for each collection, as well as a wetland classification based on established wetland classification guides provided by the US Fish and Wildlife Service. We compared data using a chi square test, which provided rough correlations between the wetland classification and the larval species collected. While this data is not complete and ideally should be assessed using a greater sampling from multiple areas, it does provide the groundwork to develop species-based habitat models using wetland classifications.

In parallel to developing a wetland-to-species correlation, our collaborators reviewed the level of information provided from multispectral against hyperspectral images when identifying likely wetlands. The hyperspectral images from Hyperion provided better wetland classification when compared to LANDSAT and moderately differing results when compared to ASTER. We believe that hyperspectral images can provide better results with new vegetation indices than can multispectral images due to the potentially relevant bands being far narrower than in multispectral imaging.

Finally, we used Hyperion images to identify specific wetlands that showed a high prevalence to *An. punctipennis*, a specific mosquito species. Using the habitat models and vegetation indices determined previously, we created a model, which allowed for a prototype remote sensing map that identified likely locations for *An. punctipennis* larva.

Significance

Insects can and have been used as bioweapons since the 1800s. It is believed that several governments, including the US, were testing insects as potential transmission vectors within their bioweapons programs prior to the Biological Weapons Convention. Insects would be a simple, yet risky, method to transmit several potential bioweapon diseases, including plague, Rift Valley fever (plus other hemorrhagic fevers), tularemia, Venezuelan equine encephalitis, and other forms of encephalitis.

Insects can also carry and spread diseases that have entered the population via a natural occurrence or by a bioweapon release of a pathogen that can be transmitted by insects, but one in which insects were not the release vector. The West Nile virus, while not a bioweapon release, shows the impact of a new arthropod-vector within a population.

Current methods of tracking and analyzing insects are very slow and labor intensive. Manual traps are placed within areas that insects are expected. The specimens are collected from the traps and tested using a number of techniques to determine the presence of a disease. The two main problems with current techniques of insect surveillance are that:

- the insects must be collected quickly and within a small insect population in order to provide an early indicator of a bioweapons release or an emerging disease
- most insect information is not shared within an international context.

Disease carrying insects do not have a wide travel range, but can easily cross borders. To enable the quick collection of insects, a better method of determining where the insects are located is necessary. In addition, information about the density of the insect population, new insects within an area, and the diseases these insects may carry must be shared with other countries within the region.

This project has created a rough prototype remote sensing model, which provides specific rather than general information on potential locations for disease carrying mosquitoes. The model could potentially be used for other arthropods as well. Such information will be important for the control of mosquitoes as well as the collection and testing of species of mosquitoes for specific disease. These diseases may be endemic, emerging, or released into a population as a biological weapon.

There is an ever-growing concern of insect vectors within the biological weapons threat, and the use of remote sensing models may be able to aid in the early detection and mitigation of these diseases. Remote sensing models can also be used across borders where ground collection of data might be politically or physically impossible. This data can then be shared with neighboring regions to help reduce disease spreading across borders.

Biological Risk Assessment Methodology (BioRAM)

79850

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Project Purpose

Legitimate bioscience facilities may be targeted as a source of biological agents for use in bioterrorism. The objective of this project is to develop a biological risk assessment computer model and methodology (BioRAM) that considers the unique challenges associated with protecting dangerous pathogens.

FY 2006 Accomplishments

We developed a methodology for prioritizing biological agents based on weaponization attributes (acquisition, production, and dissemination) and consequence attributes (transmissibility, morbidity, mortality, social/psychological impact, economic impacts, potential to become endemic). This agent-specific methodology provides the foundation for analyzing the unique assets found at legitimate bioscience facilities.

We then constructed a bioscience facility risk assessment model that includes the biological agents, other assets (e.g., information), and the threat environment. Evaluation of the threat environment includes a systematic method for analyzing the means, motive, and opportunity of potential adversaries. We incorporated this methodology into a computer software tool to enable others to conduct risk assessments of bioscience facilities.

Significance

BioRAM is designed to help a facility achieve a risk-based, graded protection strategy for its dangerous biological agents, as well as other facility assets. The risk assessment is followed by a vulnerability assessment and recommended mitigation measures that focus on reducing the unacceptable risks.

This methodology and tool will be applicable to US bioscience institutions as well as facilities throughout the world. The results of a biosecurity risk assessment will help facility management prioritize the risks the facility faces. This approach proves highly beneficial because it ensures that the level of security applied is proportional to the level of risk that exists. Perhaps an even more compelling justification for this approach is that it serves to ensure that the cost of protective measures is roughly proportional to the risk at a facility.

Laser-Induced Breakdown Spectroscopy for Remote Explosives Detection

79852
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Project Purpose

The purpose of this project is to demonstrate the feasibility of using a laser-induced breakdown spectroscopy system (LIBS) for remote detection of explosives, with future applications in robotic-based stand-off detection of large vehicle bombs. The ultimate goal is to provide a means of detecting explosives from a safe distance, thereby saving lives and reducing asset and property losses.

The project team has designed and is building a proof-of-concept LIBS system. This benchtop system is comprised of 1) a sample collection/preconcentration unit to collect and concentrate particles from surfaces being inspected, and 2) a sample analysis unit to perform LIBS detection, analysis, and identification of the collected samples.

Currently, LIBS is a highly successful technique for elemental analysis of materials, particularly for inorganic substances such as metals and metal-containing compounds. This project extends the application of LIBS to the analysis of organic substances, specifically to the detection of high explosives. If successful, this technique could be applied to other hazardous or toxic substances, such as chemical and biological agents. LIBS offers several positive attributes, particularly its high sensitivity. In addition, current LIBS systems are robust and do not require sensitive optical alignment, a benefit for field work. Other benefits include no sample preparation and rapid, straightforward analysis.

Although LIBS is very sensitive, selectivity is a problem for explosives detection. LIBS was developed for determining the elemental composition of a material; this is not sufficient information to uniquely determine the presence or absence of an explosive. Because the oxygen-to-nitrogen ratio is high for most explosives, it can be used as one of the diagnostic features for explosives. However, atmospheric oxygen and nitrogen interfere with this ratio and hinder detection. Thus, we are currently working on ways to remove the contribution from air to the LIBS signals.

Because of organic materials present, the test samples all contain carbon and hydrogen. In addition, some samples will also contain oxygen and nitrogen in varying amounts, with some samples being explosives (e.g., TNT and RDX). Unfortunately, because of the high concentration of nitrogen and oxygen in the air, we observe emission from these elements even when they are not present in the solid sample. Thus, dealing with these common interferents is critical to reliably detecting explosives. Therefore, improving the specificity of LIBS as applied to explosives detection continues to be a major objective of our overall effort.

Another part of our effort to improve LIBS specificity includes statistical analysis (i.e., chemometric analysis) of the LIBS operating parameters that we can adjust to control the experimental conditions and to potentially optimize operation of the instrument for explosives detection. In addition to statistical analysis, we are pursuing a second approach to enhance selectivity, which involves first characterizing, then controlling, the properties of the laser-induced plasma. For example, chemical reaction rates are strongly dependent upon the temperature, so controlling the plasma temperature is important.

FY 2006 Accomplishments

We accomplished all of our FY 2006 milestones.

Improved Reproducibility in Determining Sample Peak Area

Baseline fluctuations make it difficult to generate meaningful peak areas from the emission data and sometimes generate negative peak areas. Continuum effects change the baseline of the entire emission spectrum, and these effects are pronounced near the emission lines of interest.

We evaluated two ways of determining emission line intensities: 1) peak area calculation by numerical integration and 2) a peak-to-base ratio calculation using Igor software routines. These routines use a user-defined peak guideline table with peak, which defines baseline cursors settings for all peaks of interest.

We studied 100 paired-sample shots with each of the peak area routines. The peak-to-base ratio method showed significantly better statistical performance, e.g., smaller standard deviation and less scattering of the peak values for the emission lines of interest.

Designed Preconcentration Unit
Based on fluid flow modeling, we determined the LIBS preconcentration unit design by using dynamic flow software (CFDesign) for flow characteristics in conjunction with computer-aided design software (Inventor) for the components' mechanical design.

To develop the proper hardware, we performed material testing of possible substrates for material compatibility. We tested many materials with LIBS to determine the optimum sample coupon composition, while choosing materials that maintained the desirable heating and mechanical properties.

Aluminum-3003 had the least amount of undesirable magnesium emission, which obscured emission lines of interest when other materials were tested. We designed the coupon holder to reduce interferences introduced by the operator's handling. The design increased the repeatability and consistency of the sample coupon placement in the laser beam's path. We designed a locking base fixture that holds the coupon holder in place during sample collection, sample desorption, and LIBS analysis, thereby increasing repeatability and consistency.

Improved LIBS Selectivity
Selectivity of LIBS is determined by the presence and intensity of specific atomic/molecular emission lines. We believed that using the carbon swan lines and the cyanogen band head would definitively identify a nitrogen-containing compound and that the atomic oxygen and nitrogen emission lines contributed by the atmosphere could be disregarded for detection. However, identification of a nitrogen-containing compound has proved more difficult than originally anticipated.

To reduce the contribution of atmospheric oxygen and nitrogen, we will investigate the use of different laser schemes such as dual laser pulse or a femtosecond laser, which has been demonstrated to reduce atmospheric contributions to emission lines.

Used LIBS Plasma Characteristics to Improve Analysis Selectivity
Operating conditions that produce cooler plasmas have a higher potential for enhanced detection selectivity than hotter plasmas that show emission from primarily neutral atoms and from singly- and multiply-charged atomic cations. Often, a cooler plasma allows the acquisition of spectra at a longer delay time from pulse initiation. From a spectrum of the Swan band system, we are able to determine the plasma's temperature, which is close to 5700 K.

Significance
Our approach is to fully contain the laser and use a robotic platform to provide access to the sample to be analyzed. This approach allows us to eliminate eye safety issues and at the same time create a system capable of performing remote LIBS for explosives detection. This approach contrasts with other research efforts for standoff LIBS detection in which the laser is propagated in the open atmosphere and is directed at the surface to be analyzed. This approach has serious eye safety issues associated with use of its powerful laser.

Thus, the remote LIBS approach we developed could be an important partial solution to the detection of vehicle-borne improvised explosive devices. There is great interest from many sectors of the US government attempting to obtain useful equipment to deploy to our troops currently serving in Iraq.

This project will be leveraged by other Sandia explosives detection work. The information gained via fluid flow modeling of the preconcentration unit will aid our understanding of the optimum design for future explosives preconcentration designs.

Once the proof-of-concept LIBS system has been demonstrated, the next logical step for a follow-on project is to design a system capable of being located on a robotic platform. Miniaturizing the preconcentration unit and placing it on a movable robotic arm would allow the system to be positioned close to the surface of a suspect item or vehicle to collect explosives particles and vapors that would likely be present on the exterior of a vehicle contaminated with explosives.

Other important applications for materials containing unusual elemental composition (e.g., chlorine, phosphorous, and sulfur) would be of great interest to other sensor groups at Sandia.

Discrete Field-Portable Identity Microarrays

79854
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Project Purpose

The goal of this project is to develop a miniature sensing system for human identification using deoxyribonucleic acid (DNA) fingerprinting technologies with microneedle array technology. The need for real-time, selective, accurate human data collection has become a national security priority in recent years amid insidious global events arriving in this country. Determining the identity of an individual now requires samples to be transported to a laboratory where final identification is achieved within hours. We are developing a collection and sensor technology that will allow this to occur in the field and reduce processing time to 30-40 minutes.

We designed and fabricated a microfluidic package where collected mammalian cells are chemically lysed and the DNA isolated and processed for detection/analysis. The manifold is fabricated in a high-temperature, laser-curable polymer and has a footprint of 9 cm x 5 cm x 0.25 cm and moves fluids at a rate of up to 21 microliters/sec. The total volume of reagents used within the device for DNA isolation and analysis is approximately 950 microliters.

We fabricated and tested an electrode array that inserts into the fluidic manifold as a throw-away device. Complementary strands of DNA can be patterned onto the array via a diazonium chemistry developed for this application. We developed two methods for electrochemical detection of the hybridation of the sample DNA to the patterned complement. One is a displacement assay using a ferrocene mediator, while the other incorporates catalytic nanoparticles into the sensing layer. We developed a protocol that involves minimal reagents and processing for DNA extraction and enzymatic fragmentation within the microfluidic manifold.

FY 2006 Accomplishments

Develop the Microfluidic Manifold

One of the primary goals of the team was to demonstrate the collection, isolation, and detection of DNA in a microfluidic manifold. A fluidic manifold has been fabricated and tested and will be detailed in the SAND report.

Assemble the Biological Components into a Continuous Cascade

Within a fluidic sensing device, we performed extraction and enzymatic purification and fragmentation of genomic DNA from primary human keratinocytes (skin cells) and human embryonic kidney cells. We were able to reduce extraction time to less than 20 minutes, and including restriction enzyme digestion, the total protocol was achieved in a continuous manner in less than 30 minutes.

The protocol we developed for isolation and digestion of DNA prior to detection involves four simple steps: 1) simultaneous cell lysis and DNA precipitation onto magnetic beads, 2) magnetic capture and wash of the beads/DNA, 3) elution and resuspension of DNA with restriction endonuclease reaction buffer, and 4) digestion of DNA with two restriction enzymes.

Integrate the Biological Components into the Microfluidic Manifold

We initially developed and tested the above protocol in 1.5 mL Eppendorf tubes. Once the fluidic manifold was characterized and shown to successfully move fluids from one chamber or reservoir to the next, the sample protocol was performed within the microfluidic device with no operator invention.

Downstream of the reaction reservoir is the electrode array used for DNA detection/analysis. The electrode array is fabricated separate from the manifold and is a throw-away component that fits into the manifold. We demonstrated detection outside of the manifold for DNA hybridization but we have not developed the electronics on chip to perform final analysis on chip.

In FY 2005, we developed the diazonium chemistry used to pattern the complementary DNA to the electrode surface. This fiscal year we developed amplification schemes including an electrochemical displacement assay and an electrocatalytic assay using Pd coated gold nanoparticles.

Assemble a Harvesting Patch of Microposts
We studied two device architectures, designed in Foturan® and plated in nickel, gold, and rhodium while investigating device shear strength. The difference in design is the height of the needles: 300 micron tall needles designed to scratch the stratum corneum skin layer, and 450 micron tall needles designed to penetrate through the outer layer of the skin, harvesting both epidermal and dermal tissue samples.

Nonplated needles sheared with 2 mN applied force, while the plated devices sheared at forces of 50-60 mN. The 300 micron devices were tested on pig skin. Dead cells were collected in a scraping method. Even after excessive force was applied, the device remained intact with no shearing of the individual needles. We continued to use these microposts through the remainder of this project. Cells collected via the microposts are washed from the device and the collected wash is introduced into the microfluidic manifold where cell lysis, genomic DNA extraction, and final detection take place.

Significance

The development of a field portable DNA fingerprinting system capable of rapid true/false identification will put Sandia at the forefront of rapid target identification. Our technology promises an innovative approach toward anticipating threats to and preventing surprise assaults on national security through early human/threat identification.

Both of these technologies provide innovative solutions to major strategic challenges present in person-identification, which impacts defense and basic science needs of DOE. In addition, the development of new methods to decrease DNA hybridization times and thus influence rapid identification will also impact the biosensor community in bioagent identification and contribute to the development of high throughput instrumentation for genomic applications.

Integrated Optical MEMS using Through-Wafer Vias and Bump Bonding

79856

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Project Purpose

Many evolving national security applications share a common need to densely integrate arrays of sensors or actuators with their associated addressing and control circuitry. Optical microelectromechanical systems (OMEMS) are the key technology to building large micromirror arrays that will improve optical target identification capability by a factor of 3. This project will also provide Sandia with a differentiating strength in advanced free-space communication links, phased-array radar antennas, hyperspectral imagers, and chemical agent detectors.

Our approach involves the direct bonding of MEMS die to complementary metal-oxide semiconductor (CMOS) substrates via metal interconnects and matching metal bumps and will lead to arrays 10 times larger than standard packaging approaches permit. Noise, attenuation, complexity, and cost limit the size of current mirror arrays packaged by wire bonding techniques, while direct integration approaches in which the MEMS fabrication process is fundamentally altered to accommodate the electronics or vice versa suffer from compromises that reduce usefulness and applicability.

Our proposal bypasses these limitations and compromises by realizing through-wafer vias that are compatible with Sandia's ultraplanar, multilevel MEMS technology (SUMMiT™). To fully develop and demonstrate this integration process, we will leverage "standard" SUMMiT MEMS processes to fabricate and test a 4096 OMEMS mirror array and bond it to a suitable addressing/control integrated circuit.

FY 2006 Accomplishments

We made substantial progress with through-wafer vias, pixel control and drive electronics, MEMS chip-to-electronics attachment, optical MEMS array test and characterization, and overall microsystem integration. We developed a novel high-density, high-voltage (> 100 V) through-wafer via process flow and submitted a technical advance.

We started more wafer lots incorporating SUMMiT tilt-mirrors on top of arrays of poly-plug vias to support two approaches to MEMS wafer backside metallization and reductions in via impedance. We manufactured and used mirror wafers to support bonding experiments. We assessed circuit viability by comparing the footprints of various circuit approaches to the unit cell areas.

Through simulation we made substantial progress toward determining the best architecture for 125 square micron high-voltage (30 V) unit cells with an emphasis on scalability. We determined that the multiproject foundry Jazz Semiconductor (formerly PolarFab) offers the high-voltage process best suited to Sandia. We completed the majority of the work to integrate Sandia design rules with Jazz Semiconductor design rules. System level architectures, high-voltage unit cells, and control and interface electronics have all been schematically captured and simulated. The building blocks and sample array for the digitally modulated architecture were partially laid out for Jazz Semiconductor's PBC4 process.

We investigated several solutions for the bump attachment process and selected a conductive epoxy over gold bumps approach rather than a higher-temperature solder reflow approach. We made special fixtures to apply H20E-PFC, to silicon substrates with metallized pads and gold bumps. One of these substrates was bonded to an interposer and underfilled with Epotek 353ND and subsequently survived the MEMS HF-HCl release process. More work is needed to increase bonding success observed through continuity testing.

We continue to use an adaptive optics test bed that allows demonstration of wavefront correction function of MEMS mirror arrays. We developed a risk assessment and mitigation plan to avoid problems and delays at the microsystem integration level. In FY 2007 we will concentrate on the via and bonding areas, and focus on processes that will be applicable to focal plane arrays for the Grand Challenge LDRD project 95211, "Highly Pixelated Hypertemporal Sensors for Global Awareness."

Significance

While this project is focused on demonstrating the hybrid integration of an optical micromirror array and Si addressing electronics, this capability has the potential for a much broader impact. The demonstration of large, dense arrays of any type of device, such as a focal plane array, that needs individual or parallel addressing will require this capability.

Many important national security applications would benefit from the development of such a capability at Sandia, including radio frequency (RF) beamforming for electronically steerable antennas, free-space secure optical links, improved satellite imagery from the use of adaptive optics, wavefront correction of large aperture electro-optic and RF sensor systems, massively parallel optical switch matrices (or optical routers) for parallel processing, and tunable spatial-spectral filtering for remote sensing applications such as hyperspectral imaging. Other sensor applications involving flip-chip of large arrays of photodetectors to rad-hard CMOS, arrays of bioMEMS, and arrays of acoustic sensors and sources would also benefit.

This technology will not only solve the addressing problem for large, dense arrays, it will also allow for separate optimization of the drive circuitry and the optical element. Thus, compromises inherent in monolithic integration can be avoided while maximizing the system performance by maintaining advantages of MEMS micromirror devices and silicon electronics.

In addition this technology would open the possibility for integration of optical sources and detectors with MEMS structures for feedback in closed loop control of MEMS motion. Most importantly, this is a capability that will enable Sandia to integrate more of its leading edge component technologies into low-volume system solutions in a more cost-effective and timely manner than with monolithic approaches.

The development of this advanced packaging approach can impact several topics of interest, including microanalytical systems (which may use array sensors), thermal management of microsystems (by appropriate design of deep metal vias), and integrated photonic systems (at least those using array photonics). This new capability is essentially a new fabrication technology that will provide more rapid and cost-effective product realization cycle times. The need for development of through-wafer vias and flip-chip heterogeneous integration is identified in the Sandia MEMS Science and Technology roadmap.

Other applications that would benefit from our efforts include small satellites, remote sensing, chemical-biological, and nuclear nonproliferation, RF MEMS and integrated RF/photonic architectures, adaptive and tunable electro-optical and RF components for electronically steerable array antennas, diffraction limited large diameter optical/infrared imaging systems, custom sensors, beam steering without moving parts, and, of course, optical MEMs.

Stressed Glass Technology for Actuators and Removable Barrier Applications

79859

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Project Purpose

There are applications in which a material needs to serve as a barrier that must subsequently be removed. In many cases it is desirable that once the barrier has served its function that it then be reduced to small pieces. For example, in pipelines and in drilling applications, valves are needed to function as barriers that can sustain high pressures. Later, the valves must be rendered to such a small size that they do not interfere with other functions. Military applications include missile silo covers.

Other applications might require that a component be used as an actuator or as an energy storage device, and then be removed, again so as not to interfere with the function or motion of other parts. Brittle materials like glass, especially glass that is very strong or is prestressed, are ideal candidates for these applications.

Glass can be produced in different sizes and shapes. The strength and prestress, which control the fragment size, can be manipulated by either tempering or ion exchange processes. The energy stored in the residual stress profile can be harnessed to drive fragmentation of the component once it has been fractured. Energy can also be stored in the glass component in the form of stored elastic strain energy by mechanically loading it. Because of the high strength of stressed glass, a significant amount of energy can be stored that can then be released either to perform useful work or to initiate another action.

Our objectives are to advance stressed glass processing to develop prototype devices and demonstrate their potential. Work in this project included identifying and designing several prototype applications. Because complex shapes are required, we investigated technologies for bonding glass to glass, including a new low-temperature bonding process. We studied methods for releasing the energy stored in the glass, including mechanical and explosive shock initiation. And we directed our efforts toward methods for weakening the glass in selected regions.

FY 2006 Accomplishments

We conducted a review to determine what had been done previously for several potential applications of interest and prioritized applications based on potential customers' needs and the feasibility of using stressed glass. What we already knew was the performance requirements of the glass: it should have a well-defined strength; it should break into fragments with a controlled size; and it should satisfy optical or electrical requirements. Applications we investigated for stressed glass included frangible mechanical shock sensors and removable barriers. We also investigated using glass for applications in which metal is traditionally used.

One of the constraints on the use of stressed glass is that it is so strong after the ion exchange that it is very difficult to initiate fragmentation. We tested ways to weaken the glass in localized regions, including using coatings on the glass surface. We tested several types of thin films that partially blocked the ion exchange process and changed the stress profile in the glass. We used finite element analyses to model how preventing ion exchange in localized regions changes the residual stress profile.

We conducted other experiments to facilitate fracture by attaching small glass rod and tapering fiber-like protrusions to a larger and thicker glass plate using traditional glass blowing techniques. The dual section part was then ion exchanged. For the tapering fiber protrusion and plate experiment, fracture was initiated in the fiber and propagated into the plate. For the rod, fracture propagated part way down the rod and terminated. It appears that large aspect ratio shapes (length/diameter) with a constant cross section may not be able to propagate fracture over their length, whereas protrusions that are thin and gradually thicken at their attachment to a plate can propagate a fracture.

We investigated methods for releasing the energy stored in the tensile region of stressed glass using explosives. We placed the explosive directly on the flat surface or in a cavity in an ion exchanged glass plate. The glass could only be fractured when the explosive material was used in a small hole machined in the glass surface.

We used hard, sharp-tipped indenters to initiate glass fracture. We conducted fracture initiation experiments to determine the critical indentation failure load for different ion exchange conditions, for both loading and unloading failure. We also used a femtosecond laser to write lines of damage of different thickness and width inside glass plates and to initiate fracture and fragmentation.

We investigated technologies for making containers and other complex shapes from stressed glass, including machining cavities in glass and using glass blowing and molding with the glass at elevated temperatures. Because glass compositions that are ideal for ion exchange typically have high melting temperatures, it is difficult to melt or mold the glass starting with plate stock. Machining glass plate into more complex parts is feasible, but care needs to be taken to avoid creating mechanical damage and other stress risers that weaken the glass, possibly causing it to fail during ion exchange or in its intended applications.

Significance

The information learned in this project has further defined the processing and property space for stressed glass, allowing us to extend the range of applications that could use stressed glass. The work has also helped demonstrate that stressed glass can be used as part of a stress/shock sensor with directional discrimination.

Other Communications

S.J. Glass, "Glass Fracture: Can We Control It?" presented at the Frontiers of Materials Science: Student Lecture Series, New Mexico Tech, Socorro, NM, November 2005.

J. Rygel, R. Tandon, J. Glass, and M. Braginsky, "Simulation of Fracture Patterns in Ion-Exchanged Soda Alumina Silicate Glass," presented at the Fracture and Flow of Glass Conference, Penn State University, State College, PA, October 2005.

Terahertz Quantum Cascade Lasers for Standoff Molecule Detection

79861

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Project Purpose

The goal of this project is to develop narrow linewidth terahertz quantum cascade lasers to enable optical chem/bio detection capabilities for homeland defense, nonproliferation monitoring, and kill assessment in missile defense engagements.

Quantum cascade lasers (QCLs) in the mid-infrared have been successfully used in remote molecule detection with high sensitivities. Molecular spectral modes in the terahertz offer significant advantages for unique detection and can provide a separate verification for mid-infrared signatures especially in cases of overlapping signatures in a single spectral region. Terahertz QCLs have advanced rapidly since their invention a few years ago, but higher temperature operation and higher output powers are still needed for fieldable systems.

We are developing and using theoretical tools to guide the investigation of nonlinear optics employing infrared QCLs, and exploring quantum coherence to improve QCL performance, especially toward high-temperature operation. In conjunction, we are creating novel terahertz designs to increase output power. Ultimately, in addition to remote molecular and biological sensing applications, the intrinsic narrow linewidth of terahertz lasers enables their use as local oscillators for terahertz heterodyne imaging and spectroscopy.

FY 2006 Accomplishments

We expanded our theory to describe frequency mixing experiments and quantum coherence. We implemented first a bandstructure theory (8 x 8 k.p Hamiltonian), which takes into account all-important effects such as strain, band mixing, and so on. We then derived and implemented the frequency mixing theory and examined the possibilities for an optically assisted, electrically driven terahertz QCL scheme. The scheme is especially interesting for this project as it addresses the difficulties to obtain and maintain the population inversion between the subbands involved in the terahertz transition for higher temperatures. We calculated that this laser scheme not only has high power efficiency, but even shows lasing in regimes where there is no population inversion due to quantum coherence effects.

We successfully fabricated plasmon-guided 2.9 THz QCLs and experimental characterization showed continuous wave (CW) lasing operation up to 40 K. We are one of only a handful of research groups to accomplish this milestone. Also, we developed a fabrication process for metal-metal waveguide QCLs. This device geometry will allow better performance through superior mode-guiding compared to the plasmon-guided geometries. We performed process optimization in order to fabricate laser samples.

Significance

There is growing interest in terahertz technology both as an enabling technology as well as a verification/double checking technology. Potential enabling technologies of national security interest include highly discriminating chem/bio detection or explosive threat detection, high-bandwidth covert communications, and imaging applications including through-“wall.”

Sandia has a strong record in design, growth and fabrication of QCLs (in both terahertz and mid-infrared) and holds the record for the growth of the longest wavelength and highest power THz QCL to date. Potential customers have inquired about these new devices, so this project puts us in a position for follow-up funding if we demonstrate the capability to provide source development.

We are already leveraging the results to develop a much more complete subsystem in the terahertz Grand Challenge LDRD project 95214, “Terahertz Microelectronic Transceiver (TµT) System.”

Refereed Communications

I. Waldmueller, W.W. Chow, A. Gin, E.W. Young, and M.C. Wanke, “Gain without Inversion: An Approach for THz Quantum Cascade Laser?” presented at the International Semiconductor Laser Conference, Kohala Coast, HI, September 2006.

I. Waldmueller and W.W. Chow, “Influence of Radiative Coupling on the Nonlinear Optical Response of Intersubband Transitions in Multiple Quantum Wells,” presented at the Conference on Lasers and Electo-Optics, Long Beach, CA, May 2006.

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Advanced Technologies for National Security Applications

79862

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Project Purpose

Incidents like the downing of a US surveillance aircraft in China, battlefield loss of military hardware, forward deployment of sensitive assets, foreign military sales, and increasing terrorism all demand better protective technologies for military hardware and weapon systems. Recent government mandates (e.g., Gansler Memorandum) require broad implementation of protective technologies.

Concurrently, requirements for the use of commercial off-the-shelf (COTS) hardware in new military systems are increasing. Existing solutions do not provide optimal protection for systems based on COTS hardware. Thus, there is a tremendous national need for advanced protective solutions.

In this project we combined and leveraged four diverse areas of Sandia expertise and technology to create formidable new protective systems. Development of these system concepts can only be accomplished at a national security laboratory like Sandia, with expertise in microsystems, materials science software, and vulnerability analysis.

FY 2006 Accomplishments

Our development strategy focused on experiments that prove each technical element (hardware, software, and integration strategies) of our concepts. We built and tested simplified prototypes, completed successful proof of concept experiments for multiple protective technologies, and are conducting further development based on these results. In the future more sophisticated prototypes will be built and tested.

Significance

Technical challenges to implement these concepts are substantial, requiring advancement of the current state of the art in each technology area. Our team contains some of the world's experts in their respective technologies. It is clear from discussions with potential customers that success in this project could attract additional funding to mature the concepts and adapt them to specific systems. This activity also strongly complements and supports existing core capabilities required for our nuclear weapons mission.

Forensic Tool Development for SCADA Systems

79863

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Project Purpose

The purpose of this research is to identify how current forensic capabilities can be successfully applied to supervisory control and data acquisition (SCADA) systems and what critical facets of SCADA systems are not adequately addressed by existing forensic tools and methods.

FY 2006 Accomplishments

In order to facilitate digital forensics for SCADA systems, we designed and implemented extensions in the EnScript language for the EnCase digital forensic tools. We tested the use of EnCase with these extensions on actual SCADA systems in a laboratory environment and performed benchmarks to determine the likely impact they would have on an operational system.

We also developed preliminary recommendations for SCADA system providers describing features that could be included in their products to assist system owners and law enforcement with digital forensic analysis for SCADA systems.

Significance

We developed preliminary recommendations on how to safely use certain digital forensic analysis tools within SCADA environments. These recommendations will help system owners and law enforcement to monitor and protect the nation's critical infrastructure. In addition, we extended the technology of digital forensic analysis into a new specialized area of the industry.

Verification through Process Monitoring

79864

P. E. Rexroth, E. V. Thomas, B. B. Cipiti

Project Purpose

The desire to promote clean, nuclear energy across the world, and the gathering momentum for fuel recycling in the US, are requiring a renewed look at the state of the art in safeguards technologies for reprocessing. In current reprocessing plants, material accountancy is based on a physical inventory, requiring cessation of processing; this approach is often not optimized for cost or effectiveness for either the inspector or the operator.

Increased automation can allow more precise online measuring instrumentation, but it also requires a safeguards approach that can handle all of the data and provide monitoring in a more efficient manner. The goal of this work is to use automated process control and data collection for advanced materials accountancy and monitoring to improve global transparency.

We are examining advanced process control methodologies and high-precision instrumentation to verify facility processes and declarations. It is anticipated that authenticated inspector data will be required at critical processing points to validate the overall information, but the goal of this work is to minimize the deployment of inspector-owned equipment while increasing the detection limits for material diversion.

FY 2006 Accomplishments

We developed a material and uncertainty tracking model based on the UREX+ reprocessing concept. We are using this model to examine fault detection, optimize sampling strategies for both operator and inspector-owned monitoring equipment, and test the impact of new instrumentation that can reach higher levels of precision. The primary objective is to identify where instrumentation is most helpful to monitor the process and detect diversions with minimal false alarms.

The process for meeting this objective involved conducting a set of statistically designed simulation experiments that explored a wide range of operating conditions and instrument deployments. The efficacy of a particular accountancy strategy is measured by how quickly the diversion was detected (in cases of a diversion) and the false alarm rate. We analyzed the results across the range of operating conditions to assess the value of the measurement instruments (alone and in combination).

Significance

The tools, technologies, and methodologies that will result from this research will provide improved agreement verification and will result in more effective use of inspection resources and more timely detection of material diversion. Whereas the work is initially focused on nuclear processes, the resultant methodologies and technologies will be readily applicable to chem/bio proliferation concerns as well.

This work is bringing Sandia into the arena of monitoring and safeguards for spent fuel reprocessing; as a result Sandia is an active participant in the design of the monitoring system for GNEP. This work will likely lead to cooperative programs with several international partners.

The tools, technologies, and methodologies that will result from this research will provide improved agreement verification and will result in more effective use of inspection resources and more timely detection of material diversion. Whereas the work is initially focused on nuclear processes, the resultant methodologies and technologies will be readily applicable to chem/bio proliferation concerns as well.

Other Communications

P.E. Rexroth, B.B. Cipiti, E.V. Thomas, N.L. Ricker, and B.A. Purvis, “Methods for Advanced Material Accountancy: Simulation and Analysis,” in *Proceedings of the Institute of Nuclear Materials Management 47th Annual Meeting*, July 2006, CD-ROM.

High Operating Temperature LWIR Detectors for Advanced Infrared Imaging Systems

79865

J. K. Kim, P. F. Marsh, E. W. Young, J. L. Rienstra, M. S. Carroll, J. F. Klem

Project Purpose

A breakthrough in mid-infrared (mid-IR) sensor thermal management is needed to continue the resolution/sensitivity scaling, enable new classes of nanosatellites and unmanned aerial vehicles (UAVs), and enhance mission capabilities. We developed technologies for implementing mid-IR focal plane arrays (FPAs) based on InAs/GaInSb type-II strained-layer superlattices (SLS) that can operate at significantly higher temperatures than HgCdTe, thereby reducing the required cryocooler power.

The interest in SLS FPAs in the military/intelligence communities continues to build, fueled by positive developments in both the single-pixel and two-dimensional FPA performance, and SLS is most likely to replace HgCdTe in future generation mid-IR sensors. The advantages of SLS FPAs are most pronounced in long-wave infrared (for hyperspectral imaging) and very long-wave infrared (for ballistic missile defense) bands, and their single-pixel performance at > 200 K approaches or exceeds that of HgCdTe counterparts.

However, another order of magnitude improvement in detectivity is possible and required to reach background limited performance at 200 K. Furthermore, scaling up to high-resolution FPAs still requires addressing fabrication issues such as sidewall passivation for suppression of dark current. We are addressing these barriers to the acceptance and fielding of SLS FPAs.

FY 2006 Accomplishments

We invented a unique concept to address the issue of performance and resolution scaling of SLS FPAs and established the theoretical and experimental capabilities and foundations necessary for developing and promoting the technology. We made progress toward realizing the proposed device concept and characterized the first implementation. We are making further improvements and expect to demonstrate proof-of-concept in the near future.

Significance

Mid-IR FPAs that operate at higher temperatures can significantly enhance reconnaissance satellite capabilities. Our conception and development of new technologies that can realize this objective is being noticed in the intelligence community. Our success will serve to enhance Sandia's capability and role in the areas of intelligence, strategic/tactical military systems, and ballistic missile defense.

We established industry and academic partnerships to support technology demonstration. Industrial partnership could pave the way to technology acceptance and commercialization.

Tunable Dielectric Films for Frequency Agile RF and Microwave Integrated Circuits

79866

C. D. Nordquist, J. Sigman, P. Finnegan, P. G. Clem, G. M. Kraus

Project Purpose

The purpose of this work is to explore the integration and performance issues of tunable dielectric films, particularly barium strontium titanate ferroelectric films, in the context of reconfigurable and tunable radio frequency (RF) and microwave integrated circuits.

Tunable dielectric films have desirable properties that may provide performance superior to existing and emerging alternatives in solid-state or microelectromechanical (MEMS) devices. In particular, tunable dielectric films have very low power consumption compared to solid-state devices and switching speeds superior to those of MEMS devices with a relatively simple process. However, high film processing temperatures coupled with interactions with the underlying substrate and electrode materials need to be addressed prior to successful demonstration of circuits using this technology.

A key purpose of the project is to demonstrate tunable dielectric-based microwave circuits, including phase shifters, tunable filters, and tunable capacitors, to allow comparisons with existing technologies in terms of insertion loss, tuning range, switching speed, and reliability. This requires development of the first tunable dielectric-based microwave integrated circuit technology using through-wafer via holes and airbridges on alumina substrates. These circuit features are essential to a useful microwave integrated circuit technology because they allow moderately complex microstrip circuits with grounding at arbitrary locations.

Key challenges in this area include determining film synthesis conditions that will create the proper oxide while preserving the copper-tungsten via holes and developing etching approaches for the dielectrics that preserve the integrity of the underlying electrode.

An additional purpose of the project is to explore methods of film synthesis that will lower the temperature dependence of the film dielectric constant over a normal operating range. This attribute is critical for tunable circuits being used in real-world applications but must be addressed in the materials science and physics arena.

FY 2006 Accomplishments

We built upon materials synthesis and circuit design activities from FY 2005 and realized the following accomplishments.

We demonstrated the first tunable dielectric-based microwave integrated circuits containing via holes, resistors, and airbridges. This circuit demonstration included DC-20 GHz loaded line true time delay phase shifters with 33 degrees of phase shift, > 15 dB return loss, and < 1.8 dB insertion loss at 16.7 GHz, translating into a 2.8 ps/dB figure of merit. Additional time delay can be obtained by cascading these analog tunable circuits. This wafer included tunable end-coupled and combline filters.

Two separate combline filters, one at 6-8 GHz and another at 12-13 GHz, demonstrated insertion losses of 8 dB with a resonator Q of approximately 10 and roughly 6 percent tuning range in a 6 mm^2 area. Additional test circuits included end-coupled filters, reflect-line phase shifters, and interdigitated and metal-insulator-metal capacitors.

We improved the coefficient of capacitance for the tunable dielectric films from 2000 ppm/ºC to 200 ppm/ºC while maintaining loss tangents lower than 2 percent and tuning ranges greater than 2:1. We accomplished this through the use of multilayer deposition techniques tailored to take advantage of the increasing capacitance with temperature for high-barium content film in conjunction with the negative temperature coefficient for low-barium content films. As part of this effort, we explored the response and microstructure of the films as a dependence of processing temperature.

We designed and processed high-capacitance density RF and microwave capacitors and filters for low-voltage applications. Using a thinner (0.2 micron vs 0.8 micron) tunable dielectric layer allows for a higher capacitance density and tuning voltage in the range of modern low-voltage electronics (< 5 V). We processed the circuits on silica and high-resistivity silicon substrates to take advantage of the smoother substrate when compared to alumina.

We explored dry etching of the tunable dielectric films using inductively coupled plasma. Pattern definition was superior to the wet etching approach, but selectivity with photoresist and electrode materials was poor due to largely mechanical etch processes.

Significance

The accomplishments in this project represent valuable advances in the state-of-the-art in tunable dielectric film deposition and integration. Specifically, the advances in the areas of film temperature stability and integration with copper base electrodes are significant because they represent important progress toward the use of tunable dielectric films in relevant system applications. Additionally, the materials science approaches used to demonstrate these accomplishments can be applied to other problems in the area of materials synthesis.

In terms of application to Sandia's mission areas, this project has developed the knowledge and infrastructure to understand and fabricate small quantities of demonstration circuits for potential customers. The microwave phase shifter and tunable filter work is valuable for a variety of Sandia radar and communication systems, and the low-voltage, high-capacitance density capacitors may be valuable for reduced form-factor electronics in the areas of tags, miniaturized direct current and analog circuits, and other lightweight electronic systems. The integration of tunable dielectrics onto novel materials may allow integration into antenna materials to realize tunable or reconfigurable radiators, which is an area of interest to several Sandia mission areas as well as external customers.

While this project has progressed toward these applications, additional investment will be necessary to improve the robustness of the fabrication process and advance the readiness level of the technology to a point where it is suitable for system integration.

Refereed Communications

J. Sigman, P.G. Clem, and C.D. Nordquist, "Compositional Grading Effects on Permittivity Temperature Stability in (Ba,Sr)TiO_3 Films," *Applied Physics Letters*, vol. 89, pp. 132909-1-3, September 2006.

Other Communications

J. Sigman, P.G. Clem, J.J. Richardson, J.T. Dawley, C.D. Nordquist, P.S. Finnegan, and G.M. Kraus, "Effect of Microstructure on the Dielectric Properties of Compositionally Graded (Ba,Sr)TiO_3 Films," American Institute of Physics, New York, NY, October 2006.

J. Sigman and P.G. Clem, "Growth of (Ba,Sr)TiO_3 on Flexible Copper Substrates," presented at the 2005 Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2005.

Micropolarizing Device for Long Wavelength Infrared Polarization Imaging

79867

S. A. Kemme, A. Gin, S. Samora, R. Boye, T. Carter, A. A. Cruz-Cabrera, J. R. Wendt, G. A. Vawter, C. Alford

Project Purpose

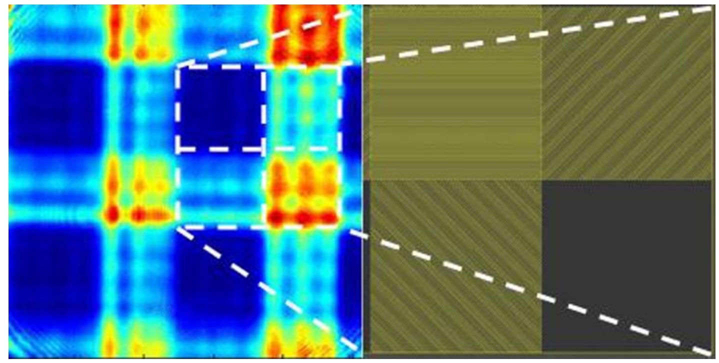
The goal of this project is to fabricate a four-state pixelated subwavelength optical device that enables mid-wave infrared (MWIR) or long-wave infrared (LWIR) snapshot polarimetric imaging. The polarization information can help to classify imaged materials and identify objects of interest for numerous remote sensing and military applications.

While traditional, sequential polarimetric imaging produces scenes with polarization information through a series of assembled images, snapshot polarimetric imaging collects the spatial distribution of all four Stokes' parameters simultaneously. In this way, any noise due to scene movement from one frame to the next is eliminated.

We fabricated several arrays of subwavelength components for MWIR polarization imaging applications. Each pixel unit of the array consists of four elements. These elements are micropolarizers with three or four different polarizing axis orientations. The fourth element sometimes has a micro birefringent waveplate on the top of one of the micropolarizers.

We fabricated the linear micropolarizers by patterning nanoscale metallic grids on a transparent substrate. We fabricated a large-area birefringent waveplate by deeply etching a subwavelength structure into a dielectric substrate. The principle of making linear micropolarizers for long wavelengths is based upon strong anisotropic absorption of light in the nanometallic grid structures. The nanometallic grid structure can be patterned with different orientations; therefore, the micropolarizers have different polarization axes.

Finally in this project, we investigated the near-field and diffractive effects of the subwavelength element apertures upon detection.



Measured infrared image (left) of 3 X 3 supercell in micropolarizer array. The individual cells are oriented clockwise from upper left: horizontally, 35 degrees, open cell, and 135 degrees, as in scanned electron micrograph of supercell at right. Input light polarization is horizontal.

FY 2006 Accomplishments

We created a reliable finite difference time domain model of four state micropolarizers or super pixels. We fabricated single-pixel-sized polarizers, with dimensions as large as 18.4 microns x 18.4 microns, to as small as 4.6 microns x 4.6 microns. The micropolarizers were made of gold on top of fused silica. We tested the single-pixel-sized polarizers and measured their extinction ratio. The measured extinction ratio ranged from 50:1 for the smallest devices to 200:1 for the largest.

We fabricated super-pixel arrays of four state micropolarizers. Every micropolarizer measured 30 microns x 30 microns. We fabricated four combinations of the four state polarizers and two sizes of the arrays for a total of eight combinations. The super-pixel arrays were made of gold on top of fused silica. We tested the super-pixel arrays by imaging the transmitted infrared intensity by the device and measured the extinction ratio between adjacent micropolarizers.

We examined diffraction effects of transmitted intensities by imaging the transmitted intensity from the super-pixel array at different planes away from the device. We fabricated a form birefringent diffractive waveplate with good achromatic characteristics in gallium arsenide (GaAs).

We tested a form birefringent diffractive waveplate across the 2-5 micron spectral region, and it performed better over a wider spectral band than any previously reported. Small errors in the duty cycle of the fabricated device led to changes in the performance, with the measured part exhibiting a total variation of phase delay rms of 9.41 degrees with an average delay of 80.6 degrees.

We simulated near-field effects between micro-polarizer and the substrate of a birefringent waveplate in GaAs. We fabricated gold wiregrid polarizers on lithium fluoride (LiF) substrate. The LiF allows the polarizer to work in the full MWIR range. We tested LiF substrate in the MWIR with extinction ratios ranging from 66:1 at 2 microns to 200:1 at 5 microns.

Significance

Imaging polarimetry is significant to the remote sensing community, and thus to Sandia, in that it can help to classify imaged materials and identify objects of interest for military applications. Traditionally, this information is gathered sequentially through a series of assembled images. Here, we employ snapshot polarimetry, where images of the spatial distribution of all four Stokes' parameters are collected simultaneously. In this way any noise due to scene movement from one frame to the next is eliminated.

For the remote sensing community, simultaneous collection of all four Stokes vectors is necessary for transient use of the gathered polarimetric signals. Moreover, this technology will allow for new signature opportunities for the overhead nonimaging infrared (ONIR) community not now available from other organizations. Finally, this work complements global efforts to integrate focal plane arrays (FPA) for advanced systems.

We quantified near-field and diffractive effects of the finite pixel apertures upon detection. We designed and built an experimental setup that models a pixel within a focal plane array to measure crosstalk from adjacent gold wiregrid micropolarizers. This configuration simulates a snapshot polarization imaging device where the two substrates are stacked; micropolarizer array substrate on top of an FPA.

Modeling and measured data indicate crosstalk between the adjacent pixels even after a few microns behind the polarizer plane. Crosstalk between adjacent pixels increases uncertainty in the measured polarization states in a scene of interest. Measured and simulated data confirm that the extinction ratio of a micropolarizer pixel in a 25 micron x 25 micron super-cell will be reduced by 30 percent when moving the FPA from only 0.5 microns to 1.0 microns away from the polarizer. These changes in extinction ratio are quite significant since typical glue separation is on the order of 10 microns.

The significance of these results is the need to increase the size of the pixels of the FPA or fold the fabrication of the micropolarizer array into the fabrication of the FPA. The first approach should reduce the effect of the crosstalk by reducing diffraction angles but increases the active area of the imaging system by factor of two or three. The second method can eliminate crosstalk but increases fabrication complexity of the FPA; however, it opens the window for further integration

Refereed Communications

R.R. Boye, S.A. Kemme, J.R. Wendt, A.A. Cruz-Cabrera, G.A. Vawter, C.R. Alford, T.R. Carter, and S. Samora, "Fabrication and Measurement of Wideband Achromatic Waveplates for the Mid-IR Using Subwavelength Features," to be published in the *Journal of Microlithography, Microfabrication, and Microsystems*.

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Electroforming of $Bi_{(1-x)}Sb_x$ Nanowires for High Efficiency Microthermoelectric Cooling Devices on a Chip

84318

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Project Purpose

The purpose of this project is to create processes to grow BiSb nanowire arrays directly on microelectronic substrates in order to develop on-chip thermoelectric cryogenic cooling. Using conventional thermoelectric device architectures, but replacing the semiconductor legs with nanowire arrays, device efficiencies can be dramatically increased. It has been predicted that the dimensionless thermoelectric figure-of-merit, ZT, for BiSb nanowires is larger than one (ZT > 1.2) at 77 K. By developing methods to manufacture and optimize such nanowire arrays, on-chip cryogenic cooling will be enabled.

FY 2006 Accomplishments

We demonstrated the ability to electrochemically grow 70 micron long BiSb nanowires with 50 nm diameters in anodized aluminum oxide (AAO) templates we developed in FY 2005. We measured thermoelectric cooling properties of BiSb nanowires by constructing an actual cooler using bulk BiSbTe p-type legs to complete the couple. ZT, the thermoelectric figure of merit, was found to be 0.12, which is nearly an order-of-magnitude below the predicted maximum. However, the BiSb nanowires used were significantly Sb-rich and had 100 nm diameters.

The measured ZT was consistent with theoretical values for this nanowire composition and diameter. The measurement was performed at room temperature and resulted in 7 °C of cooling. Theory suggests that ZT will optimize near 1.2 for 40 nm diameter nanowires with 14 atomic percent Sb compositions.

In addition, we developed advanced atomistic modeling to explore ways to lower phonon thermal conductivity, due to free surface and boundary scattering, to help determine the best crystalline and grain structures in order to optimize ZT. This is important since measuring nanowire thermal conductivity is not practical, as ZT can only be determined by building a full thermoelectric cooling device and performing measurements, which we did for one sample, and is very labor-intensive.

Instead, atomistic modeling can simulate phonon scattering in thermoelectric nanowires and enable us to study the effects of orientation and grain boundaries. We developed these molecular dynamics models and are ready to use them on realistic nanowire structures determined by transmission electron microscopy.

Significance

We developed the ability to grow AAO nanopore templates on substrates of choice that are tens of microns deep. Such AAO templates can be used for the growth of many different materials (metals, semiconductors, carbon nanotubes) to create vertical nanowire arrays for a plethora of applications.

The growth of 70 micron long BiSb nanowires by electrochemistry opens up the possibility of growing other, often simpler, nanowires for various applications that may require such long lengths.

The atomistic models for phonon scattering were actually tested for Si grain boundaries and found to agree with other corresponding published results, providing validation to our model.

Advancement in Thermal Interface Materials for Future High-Performance Electronic Applications

84320

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Project Purpose

Heat transfer across thin adhesives is becoming increasingly important as device sizes are decreasing while power usage remains roughly constant. The end result of this is ever-higher operating temperatures due to large temperature gradients established across the adhesive layer. Polymer epoxies are preferred as they are relatively easy to work with and can provide sufficient stress relief to accommodate ever-larger thermal gradients between heat source and sink.

The purpose of this project was to discover the reason for the relatively low thermal conductivity of current state-of-the-art metal-loaded polymer die attach adhesive epoxies. Measurements of effective bulk thermal conductivity with these materials typically are well below what would be predicted through acoustic theory. Manufacturers of these materials have attempted to increase the conductivity by increasing the volume fraction of the metal particles in the polymer base, but actual values are still an order-of-magnitude below predictions. In addition, continuously increasing the metal loading fraction has deleterious effects on the viscosity of the adhesive, making it more susceptible to voiding and fracture. Studies into the reasons for the lower than predicted thermal conductivities are warranted. This project attempted such a study.

We hypothesized that lack of intimate thermal contact between the microscale-sized metal particles causes conductivity values to be on an order of magnitude lower than would be predicted by standard acoustic theory. There are some data, particularly from an earlier LDRD project on wetting, that suggest that the metal particles in the polymer may be aggregating away from the two surfaces of the bond during the “squeeze” flow that occurs when the surfaces are pressed together during bonding. If true, then lack of metal-to-metal contact near the surfaces could result in lower than expected thermal conductivities. Our study intended to address this issue.

In addition, we intended to use preliminary results from squeeze flow experiments to develop a new nanoparticle augmented epoxy that resolved some of the contact issues and demonstrated increases in the thermal conductivity of the material.

FY 2006 Accomplishments

We developed a detailed thermal model of the heat transfer in a microscale-dimensioned loaded epoxy. We performed parametric analyses of the effects of packing density of the filler particles, packing style of the particles, size and density of the particles, and evaluated the effects of these parameters on the thermal conductivity of the thermal interface materials (TIM).

We investigated the bond line characteristics of these metal-loaded TIMs to determine effects of squeeze flow and viscosity on the final configuration of the metal particles. We developed a laser flash measurement process that allows determination of the effective thermal conductivity of a TIM coupon. We also performed the fixturing and preparation of these coupon samples. Ultimately, this process improves the measurement of this parameter, effective thermal conductivity, over standard thermocouple testing methods. We developed nanoscale particle candidates for inclusion into the microscale TIMs matrix. We prepared and tested representative nanoparticle-augmented TIMs coupons using the laser flash method.

We concluded from the project that the effective thermal conductivity of a nanoparticle-augmented TIM was not improved by the presence of the nanoparticles. We postulate that contact resistance between the microscale particles and the polymer filler material was not affected by the nanoscale particles, and that thermal “linking” of the two surfaces bounding the TIM (i.e., the hot source and the heat sink) was not improved as expected.

Significance

Our findings indicate that modification of TIMs with nanoparticles to increase their effective thermal conductivity presents a significant technical challenge. Although we were successful in synthesizing candidate nanoparticle sets and developing a process to include them into the microscale TIMs matrix, issues with contact resistance limited our ability to improve the overall conductivity. The sophisticated models we developed were consistent with this statement.

In addition, our modeling of microscale metal loading of polymer epoxies suggests that the size, shape, and arrangement of these particles are important parameters with respect to the effective thermal conductivity of the material.

In general, the concept of nanoinclusions to augment conductivity requires careful attention to the physics of the contact of microscale metal particles to the polymer base as well as to the nanoinclusions. Acoustic theory predicts that, should high-quality thermal contact occur, effective thermal conductivities would be an order-of-magnitude larger than currently achieved.

These results point toward future research, which should be focused on resolving the contact resistance issue and take full advantage of the modeling capabilities we developed. Specifically, the use of self-aligned nanoparticles to allow control of the orientation of them within the polymer base material shows promise for achieving the dramatic conductivity gains that are possible. One candidate concept is to use magnetic nanoparticles. If the epoxy is then cured in the presence of a magnetic field, these particles would self-align in a direction that is consistent with surface-to-surface heat transfer.

Development of Nonproliferation and Assessment Scenarios

93607

J. M. Gaudioso, D. Aceto, J. J. Larson, S. A. Caskey

Project Purpose

There are four pillars to our national biodefense program: Threat Awareness, Prevention and Protection, Surveillance and Detection, and Response and Recovery. Yet, the analyses of bioterrorism attack scenarios conducted to date have focused primarily on the last two strategic issues. To date, an analysis for the biological weapons nonproliferation community has not been conducted to identify where in various proliferation pathways the US government could best allocate resources in an effort to prevent the attack from occurring.

The purpose of this project is to develop a series of high-risk bioterrorism scenarios and then analyze those scenarios to provide a technical foundation for the first two pillars.

FY 2006 Accomplishments

We made significant progress in understanding the:

- acquisition pathways (natural sources, theft, and synthetic biology, among others)
- production methodologies (such as egg culture, batch fermentation, and cell culture)
- dissemination technologies (liquid spraying, dry powder aerosols, food contamination, and others associated with a set of biothreat agents.

We also reviewed the capabilities and motivations of known adversary groups and researched national and international biological weapons proliferation risk mitigation measures, such as international nonproliferation regimes and national legislation, regulations, and other controls.

Our research incorporates open, gray, and classified literature. We are capturing all pertinent information in a database by country, agent, and adversary. The database also includes information on specific pathogen collections, biosafety practices, biosecurity practices, and research and diagnostic capabilities of facilities that work with the subject agents.

The database was developed based on a "Wiki" structure to provide users with a familiar web-type interface to the data, to facilitate the ability of multiple members of the team to update the information, and to maintain need-to-know controls. Literature references are being managed with the software tool "Reference Manager." We also developed the scenario structure and the fundamental approach to the risk assessment.

Significance

Our research into acquisition, production, and dissemination steps and into potential adversaries yielded insights that impact many scientific and policy areas, such as those that focus on threat reduction, biosecurity, biodefense, bilateral engagement, cooperative engagement, and intelligence gathering. The research has already helped the team identify areas for future research projects.

Photonics for Ultrawideband Intrasatellite Communications

93608
G. A. Vawter, G. A. Keeler, M. E. Overberg, E. J. Skogen, A. Y. Hsu, C. Alford, G. M. Peake

Project Purpose

Next-generation remote sensing and surveillance systems focus on providing true persistent global awareness require very large focal plane arrays (FPAs) comprising many tens of megapixels operating at rates as high as 105 frames per second. This is a dramatic increase from the state-of-the-art where frame rates are limited to ~ 10 fps by available digital bandwidth for data transfer from the FPA. Improving the frame rate by using conventional technology is impractical due to the size and power requirements of each module. A new approach is needed to create a data transmission system suitable for advanced imaging systems and that is scalable to as much as 100,000 fps in a small package with low power requirements.

The purpose of this project is to develop an ultra-wideband photonic integrated circuit (PIC) for moving vast amounts of data very quickly over short distances. PICs offer an opportunity to combine into a single chip and fiber interface the capability to emit, encode, and detect multiple simultaneous wavelength channels available within the huge (> 300 THz) bandwidth of optical fiber.

We will use concepts of wavelength division multiplexing (WDM) and multilevel signaling to increase the data rate of one optical fiber to as much as 240 Gb/s. To achieve this, we will use a PIC with three wavelength channels and four signaling levels per channel. Such functionality has never before been achieved, though many of the "building blocks" do exist.

We expect that the data rate can then be scaled to as much as 25 Tb/s in just one optical fiber through use of the full dense WDM spectrum of 80 wavelength channels and eight signaling levels. This high data rate is sufficient to handle a 64 MP image at 32,500 fps using just one optical fiber.

FY 2006 Accomplishments

We established the functional configuration of the photonic circuit. The link will comprise a three-chip set for an optical carrier source, reflection modulator, and optical receiver. Using this configuration, we minimized power dissipation at the FPA end without creating unneeded complexity.

We completed designs for the wavelength-tunable laser diode, the high-speed modulator, and the high-speed waveguide photodiode. We purchased mask sets and began fabricating the first PIC pair for five Gb/s operation.

We will accomplish wavelength multiplexing using an arrayed waveguide grating (AWG). We completed the AWG designs with four optical channels spaced by 2 nm. We selected the high-numerical-aperture polarization maintaining optical fiber and purchased the optical circulator required for link function.

We also fabricated the initial PICs with lasers and electroabsorption modulators. Completion of these devices exposed several shortcomings in the grating etch and epitaxial crystal regrowth. We implemented process improvements and are fabricating new devices.

Significance

The results of this project will enable improved imaging capability of space-borne sensing systems. National users of these systems need greater image size and temporal resolution well beyond the data-handling capacity of current optoelectronics technology. Increased data capacity and reduced power consumption will enable critical sensor applications such as global surveillance against proliferant-grade nuclear weapon testing collection of critical identification information against hard targets in the modern battlefield.

Building Trusted Systems from Untrusted Components

93609

M. D. Torgerson, J. Margulies, W. E. Anderson, B. D. Kucera, M. J. Berg

Project Purpose

Typically, personal computers are assembled from an assortment of commercial off-the-shelf (COTS) hardware and software components. These COTS components are designed and produced primarily using foreign foundries and untrusted software developers. Further, the complexity of these component parts has grown to the point that they cannot be fully tested to meet functional requirements, let alone fully analyzed from a security standpoint. Even though the aggregation of untrusted components poses a serious national security concern, to date there are no viable alternatives. Such computers simply must be used in high-consequence US systems.

The purpose of this project is to develop methods of building trusted systems from untrusted components. Our efforts this year focused on studying the theoretical aspects of aggregated systems and composability, studying methods of measuring the security of the system, and examining specific technologies for building trust and actually detecting intrusions into the system.

Carefully designed security systems do prevent many types of security events. We believe it is possible to define certain, limited types of security metrics. However, the details surrounding those metrics are not yet understood, nor do we understand the value that they would bring once they are defined. Therefore, our work in FY 2007 will include determining the boundaries of what can and cannot be done with security metrics.

FY 2006 Accomplishments

We pursued two main thrust directions. We studied the theory surrounding building trusted systems out of untrusted components, and studied a particular technology, the interstitial monitoring device and produced a specific hardware instantiation of the idea, the low pincount (LPC) monitor.

We fabricated a specially designed daughter board to collect and amplify the LPC signals and send them to the LPC monitor, an Altera Stratix II field programmable gate array. The LPC monitor decodes the data, packages it, and ships it off to a personal computer for analysis. We also developed a suite of bus data analysis software and a few initial monitoring rule sets. We installed the test rule sets in the LPC monitor and tested them on a few systems.

Significance

Many people have studied the extremely difficult problem of building trusted systems from untrusted components in the past. For the most part, these efforts have yielded little, substantial fruit. A burgeoning laboratory mission is to provide technologies geared to protect our nation’s computer systems and various computer based assets. If successful, the output of this project will be a significant step forward in the theory and practice of providing security to COTS computer products. Overall, successes in this project will have the potential of providing the underpinnings for a new defining technology at Sandia.

Using Chaos for Ultrasensitive Coherent Signal Detection

93610

W. W. Chow and S. M. Wieczorek

Project Purpose

The underlying challenge of developing laser sensors, such as satellite laser threat warning receivers, is in detecting a weak coherent signal in the presence of a strong incoherent background. Demanding mission requirements make the engineering of passive detection systems, based on spatial or temporal coherence, very demanding.

In this project we explored a quantum optical solution based on the extreme sensitivity of laser dynamical nonlinearities to the coherence of external perturbation. The novelty of the idea was in the use of dynamical phase transitions to detect the presence of coherent radiation. There were many obstacles to introducing this entirely new technology. The viability of the approach depended on the detectable signal strength, achievable detection bandwidth, controllability of bifurcation conditions, and practicality of detector laser engineering. The primary goal was to understand the physics sufficiently well to either confirm the viability of our scheme or recommend alternate quantum optical solutions.

FY 2006 Accomplishments

We put in place at Sandia the necessary analytical tools for performing the investigation. The baseline scheme involved a standard Fabry-Perot semiconductor laser as the detector laser. We investigated the dynamical sensitivity of this laser to external optical signals. We performed bifurcation analysis to reveal the response of a noiseless laser to an external coherent field. We analyzed the response of a laser in the presence of spontaneous emission noise and random phase fluctuations in the external field.

Significance

The study uncovered drastic qualitative and quantitative differences in the response of lasers with different active medium parameters and laser geometry. We were able to identify the underlying physical/dynamical phenomena that determine the laser's sensitivity. Our investigation found that under specific conditions, the laser output can be destabilized by an external coherent field whose intensity is many orders of magnitude lower than the intracavity field intensity. The resulting instabilities are such that they can be exploited for sensitive detection of coherent signals.

Refereed Communications

S. Wieczorek, W.W. Chow, L. Chrostowski, and C.J. Chang-Hasnain, "Improved Semiconductor-Laser Dynamics from Induced Population Pulsation," *IEEE Journal of Quantum Electronics*, vol. 42, pp. 552-62, June 2006.

Micromechanical Resonators Applied to Shock Hardened Covert Communications

93611
R. H. Olsson, M. S. Deal, D. Ho, D. Heine, M. R. Tuck, J. E. Stevens, J. G. Fleming

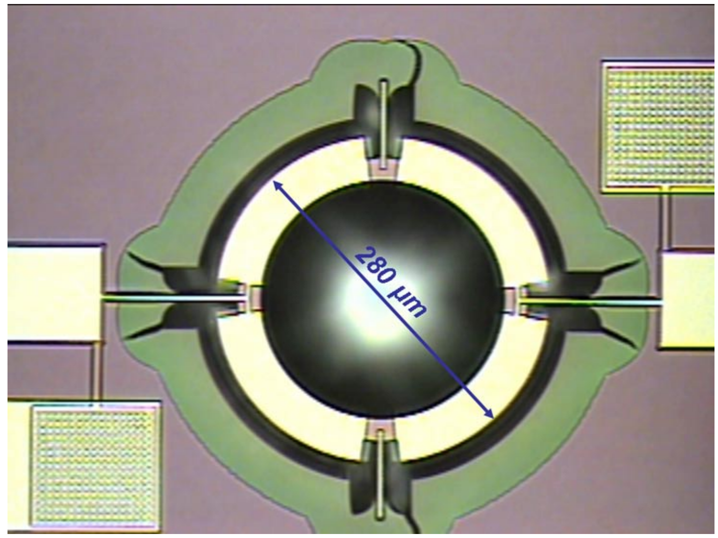
Project Purpose

The goal of this project is to develop microelectromechanical systems (MEMS) resonator-based filters and oscillators that address the needs of several national security customers. In communications systems the crystal oscillator reference restricts further size reduction. The microresonators we are developing are smaller than $1x10^{-3}$ mm^3. When compared to the smallest crystal oscillators available, 4 mm^3, this represents a significant size reduction and allows very small transceivers to be developed.

Another problem with crystal oscillators is the vibration sensitivity. The best crystal oscillators deviate in frequency by 0.1 ppb per g. While this merely leads to increased noise in most low-g systems, at g-loads greater than 100 g's, this becomes the dominant source of noise and can cause processing errors and temporary loss of a communications link. In systems like JTA (joint test assemblies) and penetrators where digital signal processing and telemetry links are expected to operate above 5 kilo-g, crystal oscillator vibration sensitivity is a significant problem.

We designed a MEMS-based resonator with a simulated vibration sensitivity of $1x10^{-5}$ ppb per g, four orders of magnitude better than the best crystals. In this project, we are attempting to verify this simulation through experiment on a shaker table and a drop table.

Another advantage of MEMS-based resonators is the ability to integrate many of them in a small space on a single substrate. This allows many high-Q filters to be used in a radio, where today the number is limited due to their large size. The final thrust of this project is to develop microresonator-based filters and radio architectures that take advantage of many mechanical filters. This thrust has applications in battle damage assessment.



AlN ring resonator.

FY 2006 Accomplishments

We developed two MEMS processes for building microresonators: a polysilicon-based process and an aluminum nitride piezoelectric MEMS filter process.

The polysilicon-based process allows for very narrow gaps (< 75 nm) between our resonator poly and electrode poly layers. This process is ideal for designing miniature, low vibration sensitivity oscillator references. We designed, fabricated, and tested an oscillator reference at 60 MHz. We are currently designing and building circuitry to sustain oscillations in the MEMS resonator.

In the aluminum nitride piezoelectric MEMS filter process, we designed, fabricated, and tested 100 MHz and 200 MHz MEMS resonators with an impedance of less than 800 ohms, and a stop band rejection of 55 dB at frequencies as high as 300 MHz. We are currently designing high-performance MEMS filter banks based on these resonators.

In addition to our MEMS resonator efforts, we also designed a five-channel transmitter and receiver that use a bank of MEMS resonators. We will be building and testing this transceiver during the remainder of this project.

Significance

The developments under this project can have a significant impact on several key Sandia mission areas. For penetrators and JTAs, the low-g sensitivity oscillators will allow data to be sent over a communications link during a high-g shock. High-speed signal processing under high-g loads is also enabled by our research, impacting devices such as smart weapons, cruise missiles, and reentry vehicles.

The small size of the MEMS oscillators and filters allows for the miniaturization of transceivers, a high priority for many groups around the laboratories. Furthermore, the high-Q of the MEMS filters reduces power consumption in communications systems, allowing scaling of the battery as well.

Finally, banks of MEMS resonators enable completely new radio architectures that were previously impractical due to the large size of other filters such as ceramic and surface acoustic wave filters. A number of Sandia mission areas are interested in this technology.

Other Communications

R.H. Olsson III, "RF Resonator Research, Development, and Applications," presented (invited) at the University of Texas at Austin, RF/Analog Seminar Series, Austin, TX, April 2006.

Infrared Detection and Power Generation Using Self-Assembled Quantum Dots

93612
J. G. Cederberg, W. W. Chow, J. L. Rienstra, M. C. Wanke

Project Purpose

Quantum dot infrared photodetectors (QDIPs) using electronic transitions of self-assembled quantum dots (SAQD) are candidates for infrared detection and power generation in the long-wavelength (LWIR, 7 to 15 µm) and very long-wavelength infrared (VLWIR, greater than 15 µm).

We are investigating the intersubband transitions of SAQD for IR sensors at the technologically important wavelength of 10 µm. Electrons in the populated conduction band of the SAQD are photoexcited to a higher energy state. QDIPs potential advantages over existing detectors lie in the zero-dimensional characteristics of SAQD: sensitivity to normal incidence radiation, increased responsivity due to increased excited carrier lifetime, and higher temperature operation due to reduced overlap of the SAQD density of states with the Fermi distribution.

The three-dimensional confinement of the SAQD modifies the symmetry selection rules, allowing the SAQD to couple to normal incidence radiation. The increased carrier lifetime in SAQD (tens of nanoseconds compared with tens of picoseconds for quantum wells) is attributed to reduced carrier-phonon coupling, termed the "phonon bottleneck." Higher temperature operation is a result of reduced overlap of the SAQD density of states with the carrier Fermi function.

Under the influence of an electric field, the photo-excited carriers are detected as a photocurrent. In spite of the theoretically predicted performance advantages of QDIPs over existing sensors, experimentally, improved performance has not been realized. The performance of state-of-the-art QDIP sensors investigated to date has been limited by the structure design, which has not been optimized for high-detectivity devices. The goal of this project is to demonstrate high-detectivity detectors that operate at temperatures above 100 K.

FY 2006 Accomplishments

We made significant progress toward demonstrating and improving QDIP single pixels. We fabricated initial "proof-of-concept" detectors and performed calibrated testing at the University of New Mexico Center for High Technology Materials (UNM/CHTM).

The preliminary devices achieved 0.5 A/W at 77 K and 4.6 V. This result represents a 30 K increase in operating temperature over previous devices that only worked at 50 K or below. The dynamic detectivity is limited by the high dark current to $3x10^8$ $cmHz^{1/2}/W$ at 77 K and 4.6 V. The possibility of having high dark currents in our devices was identified in the original proposal. This could be due to the introduction of too many free carriers into the SAQD or interaction of the dopant atoms directly with the InAs SAQD.

We evaluated design modifications and fabricated a series of structures to address both enhancements in the responsivity and reductions in the dark current. By implementing the initially proposed strain balanced design; we extended the active region thickness by a factor of five without any evidence of degradation in material quality. We still have concerns that the increased active volume will cause an increase in the dark current; diminishing the enhancement gains in responsivity will decrease the detectivity.

We calibrated the doping level introduced into the region around the SAQD and fabricated a series of samples varying electron density in the SAQD by an order of magnitude. We changed the device design to limit the exposed perimeter of the device while maintaining the optically exposed area. This should reduce the dark current without sacrificing signal. We are collaborating with UNM/CHTM to have QDIP sensors fabricated and tested. We are also completing the fabrication of these structures in Sandia's Compound Semiconductor Research Laboratory.

We modified existing theoretical models for InAs SAQD in InGaAs. We baselined our code against other results from groups at the University of Bremen and University of Kaiserslautern in Germany. This preliminary result allowed the effect of inhomogeneous broadening of the SAQD ensemble on interband laser operation to be investigated.

We obtained additional assistance from Professor H.C. Schneider at the University of Kaiserslautern. Prof. Schneider's student worked at Sandia over the summer to calculate the conduction band structure of SAQD in different semiconductor matrices. This work will enable dipole oscillator strengths of the conduction band to be calculated and compared to experimental results.

Significance

Infrared sensing has long been an important technology for military and intelligence communities. QDIP sensors can positively impact this technology by providing detectors with higher operating temperatures or with sensor functionality that is not presently achievable. Since QDIPs are fabricated in III-V materials, they have the potential to be monolithically integrated with other optical and electronic components for engineering next-generation sensing systems. These systems could be used for chemical identification enabling improved environmental monitoring and process control or thermography for electrical/mechanical fault detection.

Refereed Communications

M. Lorke, W.W. Chow, T.R. Nielsen, J. Seebeck, P. Gartner, and F. Jahnke, "Anomaly in the Excitation Dependence of the Optical Gain of Semiconductor Quantum Dots," *Physical Review B*, vol. 74, p. 035334, July 2006.

Other Communications

A. Roshko and J.G. Cederberg, "Lateral Distributions of MBE and MOCVD In(Ga)As Quantum Dots," presented at Photonics West, San Jose, CA, January 2006.

Ultrathin Ultrahigh-Efficiency Heterostructure Microcooler for Satellite Sensing Applications

93613
S. D. Mukherjee, M. C. Wanke, J. F. Klem

Project Purpose

The goal of the project is to create ultrathin coolers based on semiconductor structures to be used at low temperatures such as 90–200 K. Currently no thermoelectric coolers work at low temperatures with high efficiency, and mechanical cryogenic coolers are large and require a lot of power.

We will use a thermionic cooling mechanism, achieved by electrically forcing hot electrons in the direction of the desired heat flow. Higher energy electrons from one side of an electronic heterobarrier are selectively extracted by a small applied voltage. The hot electrons are then collected in the material on the other side of the barrier.

The deposited higher energy electrons thermalize quickly in the presence of electrons already existing in the material which, in turn, pass on the excess energy to the lattice. Thus one side loses heat to the other side and its temperature is lowered. A series of thermionic emitters would extract hot electrons at about 40-50 percent of Carnot efficiencies without mechanical vibrations. Practical implementation is limited by heat backflow and Joule heating.

This method of cooling has not been studied carefully for use at low temperatures. The main obstacle is that because the cooler is very thin, heat backflow can be very high. The thermal conductivities for almost all solids become very high in the lower end of the temperatures of interest, namely 80-100 K. Therefore, this project looks closely at the aspects of thermal conductivity at low temperatures and at how to reduce them. Success will depend upon achieving a high refrigeration-rate coupled with simultaneous low thermal backflow.

FY 2006 Accomplishments

We completed the AlGaAs/GaAs heterostructure design for a thermionic cooler. We subsequently discovered that these materials have very high thermal conductivities at low temperatures and would not work well as thermionic coolers. Therefore, we designed and grew a number of InGaAs/AlInGaAs structures that should have lower thermal conductivities and may provide better results.

We completed our fabrication plans for the devices, have the photolithographic masks, have in place our cooler efficiency estimation method using computer simulations, and made progress with our device optimization process using theoretical analysis. The simulations use classical thermionic emission theory for overall performance analyses and quantum-mechanical electron transport theory to eliminate electron reflection for increased efficiency.

As device characterization will take place inside a vacuum cryostat, we designed mechanical structures for holding samples at variable temperatures while providing a large number of electrical connections using vacuum feedthroughs. A major show-stopper for this device to perform to satisfaction is heat backflow because the device is so thin, a problem we are now in a position to address.

We consulted with Dr. David Emin, a phonon-transport and solid-state physics expert at the University of New Mexico. We devised a formulation to calculate and design broadband phonon-blockers using short-period superlattices made with dissimilar compound semiconductor layers. The method eliminates the need for lattice vibrational mode calculations that is extremely time-consuming. Our materials properties comparisons enabled us to begin to identify optimal heterostructure combinations appropriate for cooler design with phonon-blocking capability.

Significance

Passive sensing (e.g., using infrared focal plane arrays (IR FPA)) contributes to multiple satellite applications (long longevity) and ballistic missile defense applications (short longevity). Thermionic coolers are expected to be fabricated at wafer level, taking advantage of microfabrication capabilities of Sandia's Microsystems and Engineering Sciences Applications (MESA) facility. Wafer level fabrication is highly repeatable and, once reliability criteria are met, would yield high-reliability parts for satellite imaging applications.

We are also exploring the potential applications of thermionic coolers for space-based phased array radar. Altogether, therefore, thermionic coolers may contribute toward low-wattage, low-mass cooling applications for a number of National Nuclear Security Administration (NNSA) and other government missions.

The cooling capacity of mechanical cryocoolers is constrained by size and power budget and continues to be a limiting factor in the scaling of FPAs used with analog amplifiers, low-power A-D converters, and/or optical interconnects for satellite surveillance. Theory predicts that thermionic coolers may be as power efficient as 25 percent (~ 4 W/W) at a fraction of the weight, clearly superior to conventional pulsed tube cryogenic coolers that are ~ 12 percent (~ 9 W/W) efficient.

A practical arrangement would be to selectively cool the FPA and not the readout circuit (ROIC). The thermionic cooler would be in a carrier substrate on which the FPA is directly placed while electronics and optical link elements are located at the bottom or the periphery of the carrier substrate. Since this is considered highly invasive, the cooler could be placed after the ROIC.

Both the approaches would reduce weight and power requirements for conventional cryocoolers that would come after the thermionic cooler. Alternatively, the cryocooler may be replaced with passive, heat-pipe cooling. III-V semiconductor thermionic coolers offer zero vibrations and reliability in the absence of moving parts.

Wafer-level fabrication of thermionic coolers would provide high yield, environmentally sound and, once reliability criteria are met, multiple-use parts for satellite imaging applications of NNSA and DOE. Thermionic coolers, therefore, promise to be a valuable new component in the inevitable path of increased IR photodetector-electronic integration necessitated by the expanding sensitivity and resolution demands for IR imaging.

Monolithically Integrated, Backside-Illuminated Photodiode Array

93615

R. R. Kay, M. L. Holmes, S. K. Dunlap, M. Watts, J. L. Rienstra, R. D. Nasby, R. W. Young, D. J. Stein

Project Purpose

Monolithically integrated photodiodes built on the backside of a focal plane array (FPA) wafer offer density, reliability, and cost advantages over conventional hybrid sensor detector technologies. We propose to develop a novel sensor FPA consisting of silicon photodiodes built in the silicon-on-insulator (SOI) handle layer with embedded front side readout electronics. This approach offers a monolithic alternative to hybridized silicon FPAs and eliminates the need for an additional level of assembly to interconnect the detector to the readout electronics. The device structure offers advantages in assembly simplicity and pixel density. This sensor detector can be used in satellite missions requiring visible remote sensing capabilities.

Many Sandia programs in the areas of nonproliferation, remote sensing, and homeland security rely on image sensing technology. These applications additionally rely on complex signal processing to achieve their mission goals. Examples include optical transient detection and moving-target identification. Visible wavelength imaging sensors used by these programs fall into two technology categories: charge coupled devices (CCDs) or complementary metal-oxide semiconductor (CMOS) active pixel sensors (APS).

CCD technology offers nearly theoretical limited imaging performance; however, it is not suited for integration of complex circuitry needed for "smart-sensor" applications. APS technology, on the other hand, offers the ability to integrate complex signal processing with integrated photosensors at the expense of optimized image quality. The reduced imaging performance is due to the fact that the silicon has been optimized for building high-performance transistors, not photosites. Additionally, the photosites are illuminated from the front side, causing reduced fill factor since the pixel area is shared between transistors and the photodiode.

CMOS devices that are fabricated with SOI technology such as Sandia's 0.35 µm CMOS7 process offer the potential for integrating excellent imaging performance along with electronics for complex signal processing in a single monolithic device. This is achieved by creating photosensitive devices in the "handle" layer – the silicon substrate that is normally unused for device fabrication but which can be optimized for photo-optical sensors.

We propose to create an array of photosensitive pixels optimized for wide spectral and spatial frequency response. We would then design and fabricate an image-sensing device, integrating photodevices with readout circuitry, forming the basis for application-specific smart sensors. Monolithic diode integration improves product reliability, allows for higher pixel pitch, reduces overall product size, and eliminates complex packaging and cost compared to hybrid techniques. Furthermore, SOI processing offers inherent radiation tolerance to total dose and single events.

FY 2006 Accomplishments

We completed project milestones on schedule and made significant progress in the design and development of the first diode test devices.

Completed milestones include:

- Define and design the initial backside photodiode requirements
- Conduct modeling and simulation
- Develop a matrix of test structures that evaluates trade-offs in diode function, process complexity, and pixel pitch

- Develop the fabrication process
- Complete the physical design of the test structures
- Develop the custom packaging
- Test the methodology development
- Develop and fabricate mixed signal passive device capacitor and resistor test structures in CMOS7.

We completed the initial design of the diode test structures based on design specifications derived from commercial front side photodiode devices. We used modeling and simulation to evaluate device geometry, doping concentrations, quantum efficiency, absorption depth, reverse bias voltage, and breakdown voltage.

We then developed four unique process flows to implement photodiode test structures using processing options to optimize density. We used simulation results and process flow methods to create a matrix consisting of 38, 3x3 pixel arrays with variations in contact size, and pixel pitch. Pixel pitch ranged from 5-8 µm. We also architected arrays with embedded field effect transistor (FET) devices to evaluate the electrical effects of the photodiode reverse bias voltages.

Fabrication of the devices is scheduled to be completed in early FY 2007. The wafers are to be thinned to 250 µm, and backside etched to 20 µm in the diode test regions to facilitate testing. The devices will be packaged in 64 pin dual inline packages with backside test access via a laser-scribed slit. Testing will include spectral response, spatial cross talk, and frequency response.

Significance

Many intelligence programs in the areas of nonproliferation, remote sensing, and homeland security rely on image sensing technology. These applications additionally rely on complex signal processing to achieve their mission goals. Examples include optical transient detection and moving-target identification. A single chip solution combining the FPA with the pixel readout electronics provides advantages of higher pixel pitch, reduces overall product size, and eliminates complex packaging and cost compared to hybrid techniques.

SOI processing offers inherent radiation tolerance to total dose and single events. Development of this backside photodiode FPA in Sandia's CMOS7 0.35 µm SOI process offers an alternative to traditional front-side illuminated active pixel sensors designed by commercial vendors. The development of this technology will yield an integrated solution with practical pixel pitch, embedded readout electronics integration in a single die, improved spectral response, improved modulation transfer function, and superior radiation hardness compared with conventional front-side illuminated active pixel sensors. This will create a platform to integrate Sandia-developed specialized signal processing technology with high performance photosensors.

If integration of silicon photodiodes is successful, the technology may be extended to include fabrication of diodes in backside deposited thin films to extend wavelength response into the short-wave IR in a separate development effort. It may also generate interest in developing reticle stitching capability within Sandia's CMOS7 process flow to increase the size of active pixel sensor arrays, a driving requirement for many new remote sensing concepts, such as in the Grand Challenge LDRD project 95211, "Highly Pixelated Hypertemporal Sensors for Global Awareness."

Future Technology Approaches for Threat Warning Receivers

93616

S. A. Kemme, R. Boye, W. C. Sweatt

Project Purpose

Ground-based laser sources pose a risk to satellite-borne imaging systems. The level of threat can range from a temporarily blinded detector to complete destruction of the imager. There is a need to quickly identify potential laser threats by their wavelength and incident angle with high sensitivity while minimizing required package size and power.

Sandia has been a national leader in design and realization of satellite laser threat warning receivers (LTWR). Sandia technical approaches are differentiated by the ability to perform exquisite characterization of the threat laser wavelength and direction of arrival. In FY 2005, Sandia developed advanced technical concepts around next-generation LTWRs. Two of the technical concepts showed sufficient potential to warrant a more detailed investigation. In this project, we examined those two methods for identifying incoming laser radiation, incorporating either a Fabry-Perot (FP) cavity or a resonant subwavelength grating (RSG).

The FP system collects light with a wide-angle telecentric lens. The lens focuses light over a wide wavelength band through an FP filter. The FP is a resonant cavity that reflects most of the incoherent background radiation, increasing the sensitivity to laser illumination. Laser light will resonate in the cavity, resulting in high contrast fringes at the detector array. The image on the detector due to a laser input is a concentric set of two or more circular fringes. The radii and number of these fringes are unique functions of the laser wavelength, and the center depends on the incident angle of the laser.

The RSG system uses an array of diffractive elements, with a resonant reflection characteristic dependent on wavelength and angle. The RSG elements work as a tap, allowing the bulk of the input to continue through to the main imaging system while providing wavelength and angle of arrival information for input laser sources.

Long-term goals for successful LTWR systems include:

- Discriminate laser radiation from the natural background
- Improve the sensitivity by three orders of magnitude over the current systems
- Accurately measure the direction of arrival
- Determine the threat wavelength (and possibly its pulse characteristics)
- Ensure the system consumes minimal package mass, nadir-face area, and average power, with a 10 pound, 10 W goal.

To evaluate the potential of the two systems under consideration, we quantified their wavelength and angle accuracy as well as their sensitivity to laser radiation.

FY 2006 Accomplishments

We analyzed both the FP and RSG LTWRs and quantified their performance. A first-order analysis of the FP system determined several key design parameters, including the field of view of the imaging system, the etalon thickness, and the etalon mirror reflectivities. We found that the required laser irradiance for a signal to noise ratio of 1 was 1.28 nW/cm^2.

A more detailed analysis using a Matlab™ based model demonstrated that spatial filtering of the detector array output was necessary to achieve the sensitivity shown in the preliminary analysis. Moreover, high reflectivity of the cavity mirrors is required. A wavelength resolution of significantly less than 1 nm was demonstrated as well as angular resolution as low as 0.1º.

The analysis of a baseline RSG design determined several of its design requirements and performance levels. An array of RSG elements, each measuring 250 microns, could be arranged in a 40x40 pattern to provide wavelength resolution of less than 2 nm and angular of approximately 0.1º.

Significance

Sensors form the cornerstone of defensive counterspace. Sandia LTWR instruments provide exquisite characterization of incident threat lasers for their host spacecraft. In a world where laser technology is proliferating rapidly, the use of ground-based lasers against space targets is only a matter of time and opportunity. Advancement of the state of the art in LTWR is an investment the national laboratories are uniquely qualified to provide.

This project leverages and/or benefits a number of efforts at Sandia related to satellite and sensor systems. The successful completion of this project provides a quantitatively validated plan for future LTWR technology investment. This will promote Sandia's role in LTWR and answer the mandate to protect some of our nation's most valuable assets.

Shear Horizontal Surface Acoustic Wave Microsensors for Class A Viral and Bacterial Detection

93617

S. M. Brozik, T. L. Edwards, D. L. Huber, E. J. Heller, D. W. Branch, A. M. Sanchez

Project Purpose

A recent cooperative research and development agreement partnership led to successful development of a 113 MHz shear-horizontal surface acoustic wave (SH-SAW) device capable of detecting on the order of a 1000 cells/mL. This is important in suitable for medical applications where there is generally a high concentration of infectious agents present in a clinical sample. However, biological weapon agent detection concentrations are typically lower, and these devices are not sensitive enough. Detection limits as low as 10-100 micro-organisms/mL need to be achieved for leave-behind covert operations. Other sensor techniques often require additional reagents, again not practical for autonomous sensing. High-sensitivity, reagentless devices will require a more sensitive transducer and more robust stable recognition molecules.

To address these needs, we proposed to develop a miniature, SH-SAW device that operates at 325 MHz in multielement arrays for the detection of Category A bioagents in near-real time. From a technical stand point, such a detector could be used in a variety of applications, including medicine and environmental monitoring; however, we will construct a prototype bioagent detector addressing biodefense needs: monitoring facilities, waste water, and disease outbreaks. Partnering with the University of New Mexico (UNM) Medical School, we are tailoring the sensor array to detect relevant Category A agents and testing the performance against a Category A virus and bacterium using their biosafety level 3 (BSL-3) facility.

Current field-deployable systems targeting biothreats, including the Joint Biological Point Detection System, BioBriefcase, and BioWatch, are large, time consuming to operate, and require some training to use or human intervention for initial sample preparation. Our challenge is to design sensors that can provide autonomous detection of a reasonable number of bioagents in a small instrument. Successful completion of this project will put Sandia at the forefront for providing key sensor technologies for national security needs.

FY 2006 Accomplishments

Our FY 2006 milestones were aimed at developing an SH-SAW microsensor array platform operating at 325 MHz for detection of Category A bioagents. We fabricated two array designs that operate near 325 MHz on lithium tantalate. The design approach was motivated by the need to generate a pure sheer mode at 325 MHz while maintaining high phase linearity throughout the bandpass. We determined device performance by measuring a number of parameters including passband response, insertion loss, phase linearity, phase slope, and amplitude ripple. Both designs showed very good performance, and we began testing with biological molecules in UNM's BSL-3 facility.

We completed preliminary work to deposit silicon oxide waveguide layers. The thickness required at 325 MHz is about one micron. We tested two different deposition processes: a lower temperature (250 ºC) process based on silane, and a higher temperature (350 ºC) process based on tetraethylorthosilicate. Although the lower temperature process gave higher quality films, we had problems with device yield and performance. The higher temperature process resulted in films with poor adhesion to the lithium tantalate substrate and larger thermal stresses.

We are now investigating an e-beam deposition method for the waveguide layer by depositing crushed SiO_2 pellets in an O_2/Ar atmosphere, and sputter deposition using a Si source in an O_2/Ar atmosphere. These methods have a much slower deposition rate, which means only thinner (< 1 micron) films can be deposited.

The results have been promising for the e-beam evaporated SiO_2 film. Film thickness is easily controlled, accurate, and precise. The uniformity is excellent in these systems as well. The e-beam naturally heats the substrate to no more than 100 °C but still has a high residual stress in the film. Attempts to reactively sputter-deposit SiO_2 films have not been successful due to equipment failure. However, this method is the most promising of the three due to the ability to control film stress via gas pressure and power, room temperature processing, and all the advantages of the e-beam deposition process as well. Our next step will be to obtain an SiO_2 target and fix the problems of arcing and plasma instability with the system.

Four Category A agents are being produced in the BSL-3 facility at UNM. Six rounds of phage display have been performed on the Sin Nombre virus, and a candidate recognition ligand has been identified. One to four rounds of phage display have been conducted on the remaining viruses, and we will continue selection of peptides for these viruses.

Significance

This project represents an important step toward the development of sensitive, rugged, autonomous, integrated devices that can be used to manipulate, detect, and identify multiple Category A biological agents in near-real time. The success of this project would support several applications vital to national security needs in bioterrorism defense and science. The outcome of this work is production of technology for surveillance, monitoring, and detection of biological weapons.

Since we are addressing many of the challenges in a modular manner – an array for multiple detection, methods for eliminating nonspecific binding, developing regenerative surfaces, and developing new peptides for relevant Class A agents – we anticipate that relatively minor design and engineering changes could produce systems for multiple deployment scenarios such as hand-held devices, stand alone autonomous systems, or covert sensors.

In addition this device could be used in the medical community for detection, monitoring, and screening of biomarkers for infectious disease.

Post-CMOS Compatible Aluminum Nitride Resonant Accelerometers

93618
R. H. Olsson, J. E. Stevens, M. R. Tuck, E. Yepez, M. S. Baker, J. G. Fleming

Project Purpose

The purpose of this project is to develop a novel post-CMOS (complementary metal-oxide semiconductor) compatible aluminum nitride (AlN) microelectromechanical system (MEMS) resonant accelerometer. Resonant accelerometers are smaller in size, require lower power, and offer lower drift than piezoresistive or capacitive MEMS accelerometers. AlN is an ideal material for creating resonant MEMS structures because it is piezoelectric.

Using a piezoelectric drive and sense, as opposed to capacitive work, reduces the impedance and dramatically increases the power handling of the sensing resonator structure. As a result, the sensitivity of a piezoelectric MEMS resonant accelerometer is far better, particularly at low frequencies, when compared to a capacitive drive and sense resonant accelerometer.

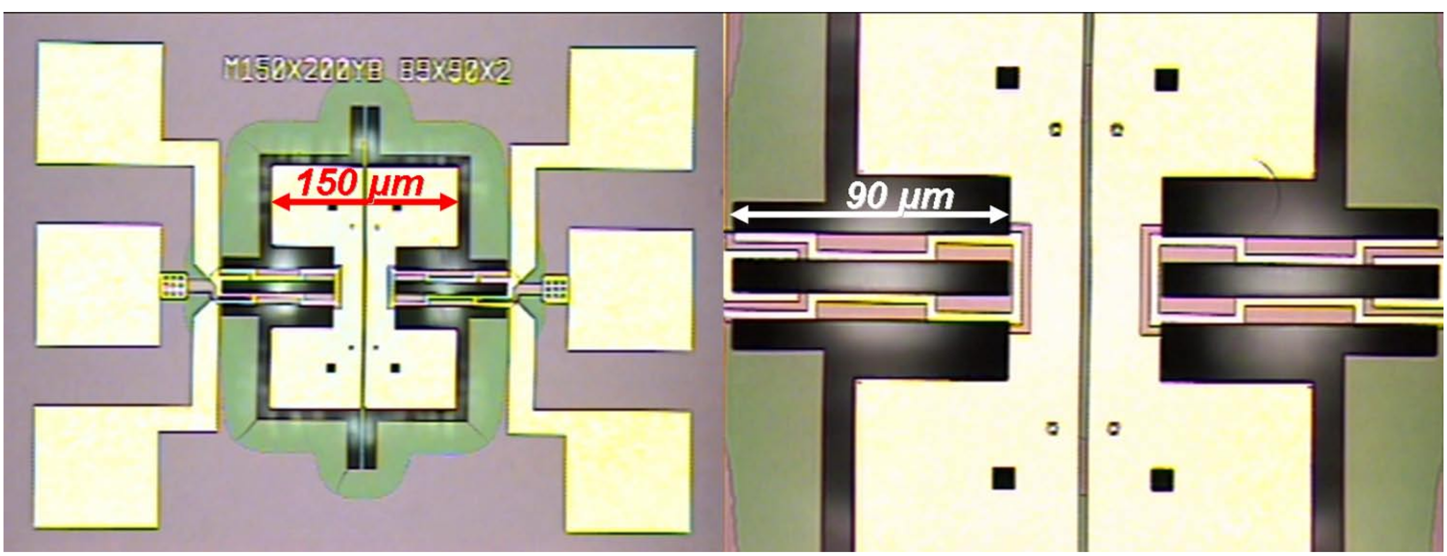
Another advantage of using AlN to form an accelerometer is that it is post-CMOS compatible. The accelerometer we are developing can be realized in a 4-mask process with a maximum process temperature of 350 °C, allowing it to be created over a preexisting integrated circuit with minimal additional cost and processing time.

In addition to developing the AlN resonant accelerometer, we are also designing low noise sustaining oscillator readout circuitry as well as drift and temperature compensation circuitry.

FY 2006 Accomplishments

We designed a mask set containing various AlN resonant accelerometers. The designs include resonant beams at 0.5, 1, 5, and 10 MHz, an array of mass sizes, single beams and double-ended tuning fork resonators, as well as single and differential electrode configurations. These devices explore the design space, trading off parameters such as sensitivity, dynamic range, bandwidth, drift, and yield. We fabricated these devices and began testing.

The first devices to be fabricated had a patterned bottom electrode near the side of the resonant beams to allow different bottom electrode materials to be explored. This patterning, unfortunately, caused a crack in the AlN film at the side of the beam where the signal transduction occurs. We fixed this by adopting an aluminum bottom electrode and extending the patterning of this electrode past the side of the beam. In the revamped mask set, we included another beam mode that resonates the beam up and down rather than



Double ended tuning fork accelerometer.

side to side. Such a beam will not suffer from the side cracking effect. These changes are in fabrication.

An effort to improve the piezoelectric coupling of the AlN layer has been under way, since the sensitivity of the final accelerometer is dependant on the coupling squared. Coupling can be measured through the orientation of the film by looking at the rocking curve full width half maximum (FWHM), where the coupling is inversely proportional to the FWHM. Original films on a tungsten bottom electrode had an FWHM of 14 degrees. We are now manufacturing devices on an aluminum bottom electrode with a FWHM of 3.1 degrees, a factor of 20 improvement in accelerometer sensitivity. Recent results using a titanium seed layer below the aluminum bottom electrode show a FWHM of 1.4 degrees. In keeping with our original goal of post-CMOS compatibility, the maximum temperature is 350 °C, and the Ti/Al stack is commonly used in our own CMOS process.

We designed a printed circuit board for low-noise readout of the MEMS accelerometers. The circuit contains two low-noise amplifiers that drive the readout beams into oscillation and a mixer that demodulates the acceleration signal down to baseband while removing temperature effects, off-axis accelerations, and drift.

We developed a piezoelectric finite element modeling capability that allows the electromechanical coupling of the accelerometers to be modeled based on electrode placement. Proper electrode placement guided by this tool gives significant increases in the sensitivity of these novel accelerometers.

Significance

The development of the described post-CMOS compatible AlN resonant accelerometer microsystem is significant to several Sandia missions. The technology also can be used to develop a number of post-CMOS compatible MEMS devices such as resonators, angular rate sensors, and RF resonators that are applicable to an even broader subset of Sandia missions.

Hand Miniaturized BW Agent Detector for Real-Time Detection of Concealed Agent Production

93619

K. D. Patel, S. Pennathur, D. Huber

Project Purpose

Obtaining accurate information on biowarfare (BW) agent production is important for the national security of the United States and our partners. Attaining the capability to remotely monitor these activities would assist in gathering time-sensitive intelligence to improve our ability to respond to such threats.

Currently, highly selective identification of biological agents in water requires several processes in which samples are collected, transported, and analyzed in central testing laboratories. This process takes several days to weeks to complete a single set of analyses. Immunologic-based (antibodies) or nucleic acids polymerase chain reaction assays require a series of complicated and labor-intensive steps for sample preparation and analysis. To overcome this problem, we propose to develop a microfabricated assay platform, which can be integrated into a field-portable sensor for detection of BW agent production.

Our approach is based on a fast, free-solution nanofluidic deoxyribonucleic acid (DNA) hybridization bioassay for detection and quantification without the need for DNA amplification. Nanofluidic devices have a distinct advantage over conventional devices in that samples are confined to regions orders of magnitude smaller, allowing for both a reduction in sample volume and a million-fold reduction in the time required for DNA to encounter its complement. Free-solution hybridization in a nanofluidic device can be used repetitively without surface fouling or nonspecific binding ideal for a remote surveillance and autonomous detection.

Additionally, hybridized DNA can be separated from nonspecific background DNA for positive detection and identification. The steric and charge interactions with the electrical double layer will provide a way to fundamentally separate hybridized DNA without the need for sieving gels or stationary phases. Coupled to a microfluidic liquid sample processor, this assay is appropriate for detection of BW agents and materials used in production in any liquid sample, including raw water.

FY 2006 Accomplishments

The primary objective for FY 2006 was to identify and develop a fast DNA hybridization assay for detection and quantification without the need for DNA amplification. Our initial strategy was to use Sandia's expertise in porous polymer monoliths to create a high-porosity scaffold to minimize diffusion lengths and hybridization times. Polymer mobile monoliths have been successfully used and functionalized to capture, concentrate (30-fold), and release 30-mer oligonucleotides.

Through kinetic measurements, we found that hybridization occurs as fast as in a few seconds with a binding efficiency near 40 percent. Limitations with nonspecific binding and variation in monolith functionalization led us to devise an alternative strategy to develop a free-solution nanofluidic system for a fast, real-time DNA hybridization bioassay.

Nanofluidic devices were first designed and optimized using FEMLAB numerical software. Channels were fabricated using conventional MEMS processing techniques in quartz substrates with nominal depths of 50, 250, and 1000 nm. Numerical simulations using our channel design suggested that for submicron-scale fluidic channels, the diffusion and hybridization times for oligonucleotides to sample the width of the channel is less than 500 µs and 500 ms, respectively. To establish a baseline, we performed initial micro-channel experiments. Results indicate that free-solution hybridization does occur in a flowing system; therefore, fluidic channels are a valid solution to the proposed problem.

In addition, numerical solutions matched experimental microchannel data to allow for quantitative measurements of hybridization efficiency and time.

Experiments in nanochannels are currently under way and are aimed to show a million-fold or greater increase in both hybridization efficiency and time.

Significance

Production of biological weapons by adversaries necessitates development of technology to obtain accurate information and irrefutable evidence. Covert and remote monitoring of waste streams from production facilities for bioagents and materials used in their production would assist us in gathering time-sensitive intelligence and improve our ability to respond to such threats.

Refereed Communications

B.C. Satterfield, J.A. West, and K.W. Hukari, "Microfluidic Polymeric Constant Flow-Through mRNA Sample Preconcentrator," to be published in *Analytical Chemistry*.

Development and Application of Quantitative Proliferation Resistance Methodologies for Reprocessing Scenarios

93622
D. H. Saltiel, V. D. Cleary, P. E. Rexroth, G. E. Rochau, B. B. Cipiti

Project Purpose

Robust and reliable quantitative proliferation risk assessment tools are critical to a strengthened nonproliferation regime and to the future deployment of nuclear fuel cycle and risk reduction technologies. Without such tools, critical vulnerabilities of processes, facilities, and systems are more likely to be missed, and efforts to strengthen the nonproliferation regime through technology development and deployment may be misinformed.

While significant development has occurred in proliferation risk assessment in the past decade, current methodologies are still hindered by foundational flaws such as over-reliance on subjective criteria, use of attribute sets that are incompatible with other methodologies, and a failure to identify, understand, or untangle dependencies existing between attributes. These flaws limit the reproducibility and the ability to review results and, therefore, the overall utility of these methodologies.

Our work proceeds from the observation that the results of applying existing methodologies cannot be reliably reproduced due to flaws in the way these methodologies assess and use both objective and subjective information. This observation suggests that creating a new methodology or attempting to "fix" existing methodologies from the top down is less likely to contribute to the field than an effort to build a stronger foundation, which may be able to improve the reproducibility of existing methodological and mathematical approaches.

The purpose of this project is to develop a limited group of attributes that expose the most critical elements of proliferation risk in a quantifiable and nonoverlapping manner. We seek to derive these attributes from directly measured inputs, while evaluating more subjective attributes separately to allow for a more transparent application of methodologies.

The goal of our research is to develop, define, and apply a short list of critical attributes for use in quantitative proliferation risk assessment methodologies. This is accomplished by identifying key proliferation risk attributes and determining their relationship to one another, developing a conceptual scheme for independently defining and quantifying these attributes, and testing the relationship between attributes using hypothetical data consistent with commercial fuel cycle facilities.

The work is the first attempt to describe the relationships and dependencies between common and relevant attributes employed in proliferation risk-assessment methodologies. Results should influence efforts to develop technology to improve proliferation resistance of current and future spent fuel treatment processes.

FY 2006 Accomplishments

We collected and reviewed every significant methodology developed in the last ten years to quantitatively assess the proliferation resistance or proliferation risk of nuclear fuel cycle facilities, systems, or processes. Using an internally-developed set of desirable methodology characteristics, we examined a subset of the most developed and widely-used methodologies to identify strengths and weaknesses and to test the initial project hypothesis that the results of applying existing methodologies cannot be reliably reproduced due to flaws in the way these methodologies assess and use objective information.

Our results confirmed the hypothesis and made clear that the establishment of a stronger foundation for methodologies would significantly enhance their value in application to a range of critical proliferation problems. Specifically, we concluded that the development of a limited set of independent critical attributes that relied on objective, measurable inputs to the greatest degree possible is the key element in the construction of reproducible metrics.

To that end, we developed and defined a set of attributes and constituent inputs applicable to fuel cycle processes. We began work on the conceptual design of quantification and independence assessment testing mechanisms for these inputs and attributes.

Significance

The goal of reducing the proliferation risk of the civilian nuclear fuel cycle has become a top national priority. Meeting this goal, however, requires that we have reliable tools for assessing proliferation risks and vulnerabilities and the efficacy of solutions developed to reduce them. These tools will also need to have the support of our international partners and be clearly applicable to large-scale commercial facilities.

The work that we have already accomplished, and that we expect to accomplish in the coming year, will add substantially to the utility and acceptability of proliferation risk assessment tools. Once strengthened, these tools can aid in:

- Identifying weak points within systems to optimize solutions
- Measuring the risk of allowing certain states to acquire and deploy certain fuel cycle systems, technologies, or processes
- Making comparisons between fuel cycle elements (deployment decisions should reflect relative proliferation risks)
- Making comparisons between fuel cycle options (open vs. closed)
- Allowing for cost/benefit comparisons between processes/technologies
- Performing cost/benefit analysis related to the addition of features, barriers, safeguards, or other tools to fuel cycle systems with the goal of reducing proliferation risk
- Determining the course for future technology research

Sandia has active programs in many of these areas that will benefit from our activity.

Refereed Communications

C.M. Méndez, D.H. Saltiel, G.E. Rochau, P.E. Rexroth, Z. Meyer, W.S. Charlton, D. Giannangeli, D. Grenèche, and S. Ng, “Strengthening the Foundation of Proliferation Risk Assessment Methodologies,” in *Proceedings of the Institute of Nuclear Materials Management 47th Annual Meeting*, July 2006.

Collaborative Situational Awareness in Network-Centric Warfare

93623
J. H. Ganter and L. A. McNamara

Project Purpose

The purpose of this project is to understand how network-centric warfare (NCW) amplifies and diversifies mental teamwork in national security operations. The project uses applied models of cognition (mental information management) and groups (shared identity, priorities, and information) to investigate how teams grasp implications from data, form and debate alternative explanations, and plan further data collection, analyses, and actions.

The specific focus is NCW mission situational awareness, a shared real-time conception of goals and context that adapts joint action so it remains effective and valuable – despite uncertainty, change, and surprise. NCW sensor data and collector-analyst-shooter connectivity fuels mission situational awareness that is fast, insightful, and usable in some circumstances but not others. Understanding how this process works (and why it sometimes fails) will be a significant and timely contribution to the theory and practice of national security systems engineering and management.

FY 2006 Accomplishments

- We developed a comprehensive study protocol to protect and anonymize human subjects within network-centric warfare work environments.
- We conducted over 150 hours of immersive field observations and interviews inside NCW information operations, including different technical disciplines, functional teams, and crew positions.
- We learned to observe and document the data flows, behaviors, and communications within and between both formal (colocated) teams and ad hoc (virtual) teams (collaborations).
- We discovered that individuals and teams thought to be situational awareness consumers were also acting as producers, that very small amounts of verbalized information had large system effects, and that roles (such as brokers) and joint actions emerge spontaneously without explicit command and control.
- We published a report describing the teamwork observed and initial interpretations. This report has drawn interest because it shows an integrated picture of not just flows of data and commands, but awareness and improvisation.
- We began to investigate the unexpectedly continuous and terse multiparty dialogue (conversations) in NCW operations. We are exploring the psychological and functional neurological basis of this situational dialogue.

Significance

We showed that immersive independent observation, informed by theory from analogous domains, can reveal operations processes and innovations that are unrecognized or unarticulated by operational personnel, their managers, and engineering/technology providers.

Generalized, readable analyses based on these observations are helping NCW operations managers to understand their human and technology capital, evolving missions, new work practices, rerouted data flows, and informal work partnerships (collaborations) that cross organizational boundaries. Neutral analyses provide unique evidence to these decision makers as they weigh organizational and technological investments and initiatives.

More detailed analyses based on observations will help NCW operations and maintenance engineers to understand how monolithic systems are being connected and combined in unanticipated (and minimally documented) ways. Engineers will gain insights into deep requirements and technology gaps that may inspire or customize new technology offerings for rapid acceptance and customer benefit.

Multispectral Fusion for Beyond the Fence Intruder Detection and Assessment

93628
C. L. Nelson, J. J. Carlson, B. D. Nelson

Project Purpose

Expanding the envelope of detection, assessment, localization, and tracking to distant adversary activity beyond fixed perimeters, as well as inside the fence insider activities, is extremely difficult due to uncertain dynamic and harsh environmental conditions and to highly skilled adversaries employing charge coupled device (CCD) techniques to evade detection.

Current sensing technologies used in security applications are capable of detecting distant activity but are typically plagued by large amounts of noise, creating a high percentage of false alarms, and they do not provide intelligent assessment/characterization, localization, or tracking capabilities. Visible and infrared (IR) spectrum two-dimensional (2D) and three-dimensional (3D) vision systems that are used to detect intruders and unauthorized activities do not perform satisfactorily in harsh (e.g., fog, snow, camouflaged) environments or those with significant lighting variations. These performance aspects make it difficult for operators to quickly and positively assess long-range detections before the boundaries of the perimeter are reached.

New commercial multispectral cameras, low-cost uncooled long-wave IR cameras, and high-speed ultraviolet cameras provide new opportunities to conduct research and to develop new multispectral 2D and 3D sensor algorithms. The development of new multispectral sensing algorithms will enable automated sensor-based detection, assessment, localization, and tracking in harsh dynamic environments. By fusing different spectral passive imagery, the fidelity of sensed activities is increased, resulting in opportunities for greater system intelligence for inferring and interpreting these activities and formulating automated responses. This project will focus on cross-spectral and spatial image fusion algorithms to leverage unique characteristics provided by each spectrum being sensed. The results of this project will provide not only enhanced detection, but also automated assessment capabilities with results that are immediately available for timely and intelligent response actions.

FY 2006 Accomplishments

We obtained multispectral data both for co-aligned sensors and sensors that are not co-aligned with the use of electronically tunable interference filters that cover three spectral bands: visible (VIS), near-infrared (NIR), and short-wave infrared (SWIR).

We also began to develop theoretical foundations and algorithms for processing images from different spectral bands to determine how best to combine them. We looked at combining them at the 2D data level, whereby registered images are merged together to bring out the salient features of each spectrum. We also developed an initial approach to combine the images at a higher level in which each image is mapped onto a common 3D coordinate frame. This allows us to perform 3D fusion of imagers that are not co-located. We will continue to develop this approach next year.

We also developed a framework for the fusion system that will allow various types of data to be entered into the fusion engine. Application-specific algorithms will be able to access and process the data and create generic objects that can be easily handled by the operator end of the system. This framework is a significant step toward the assessment capability that the alarm, communications, and display system needs to insure the success of virtual perimeter-type architectures that rely on networked sensor capabilities.

Significance

Our work has strong relevance to the national security missions of Sandia's Defense Systems and Assessments and Homeland Security and

Defense strategic management units. The successful development and application of this technology will enhance levels of security for US forces. We anticipate that the results of this project will be useful to a specific application for an NNSA funded project. Our work is also helping to guide the approach taken on other current projects using 3D sensing. The multispectral capabilities we are developing should provide dramatic improvements in both detection and assessment for military, security, and intelligence operations.

Novel Design for Improved Nuclear EMP Detection

93629

W. G. Breiland, A. E. Mihalik, H. A. Smartt, R. M. Axline, P. L. Dreike

Project Purpose

NNSA has a long-standing mission to develop sensors to monitor nuclear explosions in support of treaty verification and war fighting missions. Our project aims to explore novel methods for improving the detection capability of electromagnetic pulse (EMP) radiation that is released from nuclear explosions.

FY 2006 Accomplishments

We proposed and analyzed two new detection schemes; both offer a significant improvement over conventional EMP sensors. The most sensitive design pushes the limits of current space-borne computational capabilities and may be difficult to implement with present-day technology unless we can develop a more efficient algorithm in FY 2007. The second design is slightly less sensitive, but requires far less computational overhead. It is highly likely that this design could be implemented as an improved space-borne EMP sensor.

Significance

An improved space-based EMP sensor would enhance our ability to detect proliferant or clandestinely tested nuclear weapons. This is a central mission of NNSA's Office of Nonproliferation Research and Engineering. Once these novel designs are fully explored and vetted, they could become part of the next generation of satellite sensors.

Adaptive Antenna Tuning for Miniaturized Tag Transceivers

93630
C. W. Ottesen, R. R. Halle, B. C. Brock, D. K. Steele, W. E. Patitz, J. R. Griffin, J. J. Thomson, J. A. Payne

Project Purpose

The purpose of this project is to develop an adaptive antenna tuner for miniaturized radio frequency (RF) tags that in practical applications will enable a 10 X improvement in effective tag transmit power and receive sensitivity.

As the size and power requirements of RF tag transceivers continue to shrink, the demand for ever smaller antennas increases. Invariably, applications that demand miniature tags present physical surroundings that detune their small, high-Q antennas, effectively reducing the tag's operating range. Increasing the transmitter power and receiver sensitivity to "buy back" range performance increases the tag power consumption, complexity, and size.

We propose using the tag's transmitter and receiver together as a measuring device to sense how well the antenna is tuned. We will stimulate the antenna with a small amount of transmit energy and simultaneously measure the energy reflected by the antenna using the tag's receiver. This reflected energy is then minimized by tuning a reactive element in the antenna.

We will develop several miniaturized tunable antennas using a solid-state or a microelectromechanical system (MEMS) tuning element. We will also develop a miniaturized antenna-sensing element that can be integrated "on-chip" as part of the tag transceiver. Finally, we will develop an adaptive tuning algorithm that can be implemented with low-powered micro-processors that are available in candidate tag systems.

If successful, this project will enhance the capability of present and future Sandia long-range tag systems developed for the US government by allowing greater freedom in tag concealment. This will be accomplished without sacrificing range performance or appreciably increasing the size of the tag.

FY 2006 Accomplishments

We identified several tags that could benefit from the inclusion of an adaptive antenna tuner, noting their operational frequency, typical use environments, and what existing resources are available in the tag to implement the antenna tuner, i.e., component capabilities, power, and volume.

We developed and tested a miniature tunable PIFA (planar inverted F antenna) using solid-state switching elements and are investigating a miniature tunable MLA (meander line antenna) that would work with these tags.

We investigated several antenna match measuring concepts including using a miniature circulator, a miniature directional coupler, a half-bridge, as well as using a zero-IF receiver that may already be in the tag. Experiments using the circulator and directional coupler have been successful in measuring return losses in the 0 to -20 dB range. Unfortunately, these parts are not easily incorporated on-chip. The smallest coupler we have found to date is 1.6 x 0.8 x 0.55 mm at 825 MHz.

Experiments with several typical Gilbert-cell zero-IF down-converters and receivers have failed. These systems were optimized to minimize local oscillation (LO) leakage out of the RF port and hence are not well suited for the antenna measurement task. In systems where the LO-RF isolation is not as good, the return loss measurement may still be possible.

We investigated several ways to provide the stimulus to measure the antenna. A very simple solution is to use a noise diode. Unfortunately, it requires a higher DC voltage and power than desirable.

We also investigated transceiver integrated circuits that have their local oscillator signal accessible. We would couple a small amount of this signal and modulate it to produce the desired antenna stimulus. This is very attractive since it would require virtually no extra power and a few parts that would fit in an area of 1.5 x 2.0 x 0.55 mm to implement. The parts could easily be incorporated on-chip in the future.

We developed a gradient search algorithm using a simple microprocessor typically used in a tag that will null a DC voltage. This algorithm will be modified to tune the antenna when the rest of the brass-board is defined.

We developed an automated capability to present different loads to the measurement circuit to facilitate the automatic tuner system development and characterization.

Our next step is to incorporate all these pieces into a brass-board to facilitate development of the adaptive antenna tuner system. Once the brass-board is functional, we will begin integration of the adaptive antenna tuner into the tag to determine what operational improvement we can achieve.

Significance

Sandia has many years of experience in providing systems to gather and exfiltrate data to various government customers. There is constant pressure to make these systems smaller and place them in various uncontrolled local environments. RF tag technology is ideal for these applications and is receiving a lot of attention from these customers.

The adaptive antenna tuner should allow more latitude in RF tag placement still close the RF link, allowing the application of these miniature RF tags to a whole new class of data gathering and exfiltration problems.

Electrochromic Adaptive Optics for Novel Functionality of Earth-Staring Systems

93631

D. R. Kammler, J. D. Blaich, J. C. Verley, E. J. Heller, W. C. Sweatt, W. G. Yelton

Project Purpose

Electrochromic (EC) materials have been incorporated into numerous devices like "smart" windows and mirrors. When a potential is applied across an EC stack, reversible processes give rise to changes in refractive index and absorption in the film stack. We believe the simultaneous changes in refractive index may allow creation of large-area Fabry-Perot (FP) filters.

Ultimately, an FP filter with a transmission peak centered at 600 nm with a FWHM (full width at half maximum) of 1 nm, a transmission of at least 30 percent at 600 nm, and a diameter of 4 inches is desired. Our goal is evaluate whether an EC-FP filter can achieve these objectives. We previously demonstrated the concept of an EC-FP filter by sandwiching an EC stack between two thin gold reflectors/electrodes. This crude device had an FWHM of 50 nm or more, and could not be modified significantly because the top electrode had to be a thin gold layer that allowed transport of atmospheric humidity into the device to make it function.

We plan to:

- Develop a process for a Ni oxide/hydroxide charge storage layer to eliminate need for atmospheric humidity and allow more complex film stacks to be constructed
- Reduce the FWHM and increase the intensity of the peaks in the etalon output spectrum by developing dielectric reflector stacks with high reflectance and low absorption
- Evaluate the extent to which it's possible to tune an EC-FP filter operating at 600 nm
- Evaluate the potential of incorporating EC materials into other devices such as a spatial light modulator.

FY 2006 Accomplishments

Using both experimental methods and modeling, we evaluated the potential for EC materials to be incorporated into an FP filter to allow modest amounts of tuning. We used a combination of chemical vapor deposition (CVD), physical vapor deposition (PVD), and electrochemical methods to produce an EC-FP film stack consisting of an EC $WO_3/Ta_2O_5/NiO_xH_y$ film stack (with indium tin oxide electrodes) sandwiched between two Si_3N_4/SiO_2 dielectric reflector stacks.

We developed a process to produce an NiO_xH_y charge storage layer that freed the EC stack from dependence on atmospheric humidity and allowed construction of a complex EC-FP stack. The refractive index (n) and extinction coefficient (k) for each layer in the EC film stack was measured between 300 and 1700 nm. We produced a prototype EC-FP filter that had a transmission at 500 nm of 36 percent and an FWHM of 10 nm.

We developed a general modeling approach that takes into account the desired passband location, passband width, required transmission, and EC optical constants in order to estimate the maximum tuning from an EC-FP filter. Modeling shows that minor thickness changes in the prototype stack developed in this project should yield a filter with a transmission at 600 nm of 34 percent and an FWHM of 9.4 nm, which could be tuned to 598 nm with an FWHM of 12.3 nm and a transmission of 16 percent.

Additional modeling shows that if the EC WO_3 was optimized, a shift from 600 nm to 598 nm could be made with an FWHM of 11.3 nm and a transmission of 20 percent. If (at 600 nm) the FWHM is decreased to 1 nm and transmission maintained at a reasonable level (e.g., 30 percent), only fractions of a nm of tuning would be possible with the film stack considered in this study. These trade-offs may improve at other wavelengths or with EC materials different than those considered in this study.

Finally, based on our limited study, the severe absorption associated with the refractive index change (in the EC materials in this study) suggests that incorporating EC materials into spatial light modulators would allow for only negligible phase correction before transmission losses became too severe. However, other EC materials may allow sufficient phase correction (with limited absorption) to make this approach attractive.

Significance

Applications of electrochromic materials tend to leverage their tunable absorption. Our application leverages the associated change in refractive index. To our knowledge, there is no detailed study that examines the feasibility of incorporating EC materials into adaptive optics like the EC-FP filter.

We demonstrated that EC materials can be incorporated into FP filters and provided a model for filter designers to use in order to evaluate whether an EC-FP filter could meet their needs. Such a filter could make possible temperature drift compensation with a light-weight power-efficient system that is compact and inexpensive.

New Hash Function for Data Protection

93633

R. C. Schroeppel, S. A. Caskey, T. J. Draelos

Project Purpose

Hash functions play a vital role in information surety, as integrity checks, and as part of digital signatures. They are used in authentication processes that provide integrity of data that is collected in untrusted settings for monitoring nonproliferation agreements. Recent cryptanalytic successes have broken older hash functions and are threatening the National Institute of Standards and Technology (NIST) standard secure hash algorithm (SHA-1).

Consequences of the breaks have included papers showing how to substitute malicious code for innocent while preserving the hash and signature values. Almost all hash functions in use have been derived from the MD4 (Message Digest 4) hash algorithm. Consequently, new ideas for hash designs are needed. We are developing a new hash function that is bullet-proof against known problems and has sufficient security margin to foil future attacks.

FY 2006 Accomplishments

We met our FY 2006 milestones and are working on the next steps.

We made a detailed review of how the problems with SHA-1 interact with IAEA (International Atomic Energy Agency) authentication methods. The review confirmed the desirability of switching to a new hash function. To assess the urgency of replacement, we are monitoring further cryptanalytic progress, by the public crypto community, against MD5 (Message Digest 5, an older standard hash algorithm), and SHA-0 and SHA-1.

We developed a software tool for assisting a crypt-analyst in analyzing weaknesses of hash functions. We designed mitigation measures for the known attacks and the identified weaknesses of SHA-1. An initial implementation of the mitigation measures is nearly complete.

We converted existing block-cipher measurement software to measure hash function quality. We developed a collection of ideas for improving on the basic SHA-1 design and are pursuing a few alternative tracks, ranging from minimal repair through major overhaul to complete replacement.

We benchmarked several existing alternative hash functions to get an idea of what performance penalties are necessary and tolerable for prospective SHA-1 replacements. We are looking at public field programmable gate array (FPGA) implementations of SHA-1 to see how hardware cost and complexity are affected by prospective SHA-1 patches, and have created our own FPGA implementation of SHA-1 to experiment with.

Significance

Our investigations confirmed the necessity to replace SHA-1 promptly in authentication and signature applications and to consider it obsolete for most other uses. We identified several promising improvements to SHA-1 and several alternative design ideas for a new hash function. When completed, our improved hash function should find wide use in many communication systems.

In light of the seriousness of recent hash attacks, NIST may initiate a competition to create a new standard hash function much like they did in creating the Advanced Encryption Standard (AES). If we develop a successful algorithm, we will be a good candidate for this standard and can influence other designs.

The IAEA is concerned with the integrity of safe-guards data and therefore requires the use of authenti-cation on all remote and unattended monitoring systems as well as for use in transmitting reports over a public network. IAEA currently requires specific use of the SHA-1 algorithm for all data authentication. If the SHA-1 algorithm cannot be used, another algorithm must be approved for use within the specific deployment. The international community, including IAEA, will likely follow the recommendations of NIST, which will, in turn, affect technical work at Sandia.

Refereed Communications

E. Anderson, S. Caskey, T. Draelos, A. Lanzone, W. Neumann, R. Schroeppel, K. Tolk, and M. Torgerson, "Impacts of Collisions within Hashing Algorithms and Safeguards Data," in *Proceedings of the Institute for Nuclear Materials Management, 47th Annual Meeting*, July 2006.

Borazine Precursors for Boron Nitride Antifriction Coatings for MEMS

93636

T. T. Borek III, R. K. Grubbs, M. T. Dugger

Project Purpose

The purpose of this work is to develop molecular and preceramic polymeric precursors that will lead to antifriction coatings for microelectromechanical system (MEMS) devices.

Boron nitride may be produced from molecular and preceramic inorganic polymer precursors. The intent is to develop molecular precursors that will permit conformal coatings of boron nitride, a solid-state lubricant and electrical insulator.

FY 2006 Accomplishments

- Established infrastructure to synthesize borazine molecules. These molecules are air and moisture sensitive and require specialized glassware, solvents, and a glovebox for handling
- Obtained all administrative approvals to synthesize the molecules of interest. Obtained laboratory space to perform synthesis
- Established analytical and characterization framework to support molecular and materials synthesis Performed preliminary coating experiments using atomic layer deposition on a variety of substrates. These films are being characterized for film composition and quality.

Significance

The results of this project may increase the lifetime of MEMS devices.

Refereed Communications

M.A. Rodríguez and T.T. Borek, "2,4,6-Tris(dimethylamino)-1,3,5-trimethylborazine," *Acta Crystallographica Section E*, vol. E62, pp. O3341-O3343, August 2006.

Remotely Interrogated Passive Polarizing Dosimeter (RIPPeD)

93639
S. A. Kemme, R. Boye, S. Samora, C. M. Washburn, D. R. Wheeler, S. M. Dirk

Project Purpose

The purpose of this project was to design and fabricate conductive polymers composed of long carbon chains, which were configured for optical performance as polarizers. Remote detection of radiation is a difficult problem due to the $1/r^2$ fall-off. Recent advances in polymer research and nanoscale fabrication methods along with advances in optical polarimetric remote sensing systems suggest a solution. The long-term goal is to fabricate a passive radiation dosimeter out of a polymer no thicker than a clear piece of tape, which may be affixed to an interior or exterior surface.

Under hard (e.g., neutrons, beta) radiation, the polarizing response of this material will change in a predictable way, which can be then observed, tracked, and measured by a remote polarimetric sensor. For example, a reflected optical beam would become less linearly polarized with cumulative radiation dose. Pairs of these dosimeters (control/experiment) may be employed to cancel background radiation effects. Moreover, it may be possible to vary the integration time window of the polymer tape to isolate signals of interest from ambient radiation.

This project will leverage the radiation-electric polymer materials already developed and patented by Sandia. Remote-sensing instrumentation will consist of both active and passive visible through near infrared (VNIR) polarimeters and passive thermal infrared (TIR) polarimeters. This polymer work differs from previous work in that the targeted material response is purely photonic, and thus will be remotely interrogated with an optical signal.

FY 2006 Accomplishments

We made significant progress on the milestones for FY 2006. We investigated the effect of polymer conductivity on the required geometric parameters and the expected transmission of an infrared polarizer. The final design of the polarizer will be done when the material development can provide the appropriate parameters. We also did considerable work on the development of polymer deposition and patterning processes.

Investigation of the original target polymer, D1PPV (poly[2,5-bis(3’7’-dimethyloctyloxy)1,4-phenylenevinylene]), revealed several shortcomings including the need for chemical doping in order to achieve good conductivity, decrease of conductivity with time, insolubility, and difficulty producing thick films.

These issues led us to pursue three independent approaches for making defined gratings of organic conductors. The three processes are a subtractive process, an additive process, and direct photo-patterning with ultraviolet (UV). We successfully developed a method to coat multilayer films for the subtractive process to provide the thickness (> 1 micron) required for an IR polarizer. In addition, the coating process includes crosslinking steps that make the polymer robust enough for patterning with traditional photoresist processes and subsequent etching.

The additive process relies on known electrochemical techniques to grow conducting polymers from solution. We grew polymerized conductive films on metal substrates and built a new electrochemical cell specifically to accommodate growing films on silicon.

For both the subtractive and additive processes, we achieved greater than one micron thicknesses as well as greater than 100 Siemens/cm for conductivity. Greater thicknesses and conductivities allow for optimal extinction ratios of the polarizers.

Based on preliminary work on a photodefined grating, we identified a candidate material with the potential for good conductivity in an environmentally stable polymer. The optical characterization of the polymers includes measurements of conductivity and surface roughness made during the development of the polymer process.

Significance

This project is designed to develop and test a radiation-sensitive passive dosimeter polymer with very low optical visibility (as innocuous as a piece of tape.) The RIPPeD tags we are developing will be useful in three broad areas:

- Interior facility monitoring and surety
- Remote exterior facility monitoring
- Tagging, tracking, locating (TTL) and monitoring vehicles/containers

Of these three applications, TTL applications of the polymer do not require radiation-sensitivity and hence are more direct applications of low-visibility polarized polymer materials.

Other Communications

R.R. Boye, S.A. Kemme, D.R. Wheeler, S.M. Dirk, S. Samora, C.M. Washburn, M.L. Thomas, "Submicron Patterning of Conductive Polymers for Use in Infrared Polarizers," to be published in *Organic Photonic Materials and Devices IX*.

New Approaches to Addressing the New Design Basis Threat

93641

M. K. Snell, K. W. Mitchiner, P. B. Merkle, J. H. Whetzel, J. Milloy

Project Purpose

The purpose of this project is to develop a systems approach for evaluating extended detection/assessment systems that matches up current new approaches for modeling the threat with security modeling and simulation approaches currently in use or under development.

Early warning or extended detection/assessment systems (EDSs) consisting of radar, thermal, lidar, visual sensors and assessment devices, as well as networks of small low-cost wireless sensors, pose significant problems for defenders as well as attackers. Significant sums are being invested in extended detection systems with little knowledge or evidence that it will or can help with the overall security of a facility. This extended detection capability, being new, requires a systems engineering approach for proper integration into the overall security "system."

This approach needs to address the following issues (among others):

- What is the basic operational philosophy for such systems, given the individual limitations of the components?
- How would an adversary group attempt to defeat or bypass such systems? Also, how might such systems complicate the adversarial planning process?
- What are the fundamental performance metrics associated with extended detection, in terms of security effectiveness, reliability, nuisance/performance alarm rate, operational effectiveness of the facility surrounded by the system?
- How can this information about operational philosophy and the threat be combined into metrics – especially performance metrics – for the overall security system.

FY 2006 Accomplishments

We researched and then selected different philosophies for creating EDSs keyed on defending against one or more adversarial approaches to the site, e.g., walking/running, or using some sort of vehicles. The operational philosophy is also influenced by how the response will use the resulting information tactically.

The adversarial perspective is very important in studying the strengths and limitations of EDSs. We believe that EDSs can be very effective by influencing where an adversary planner will want to attack and by presenting uncertainty about security features before the adversary attack will even have entered the traditional perimeter intrusion detection and assessment system (PIDAS). To try to confirm this hypothesis, we developed a:

- risk assessment methodology – the adversary/defender modeling grammar – that incorporates three coupled equations: security risk, adversary utility, and frequency of attack. These equations can also be viewed as metrics from a multisided game.
- detailed model of a generic adversary planning process to explicitly identify where EDSs can complicate or lengthen the planning process.
- specialized grammar to accurately record what is both in the attack plan and in the protective force response plans. We believe this grammar can be connected with existing software to improve scenario plans used in vulnerability assessments.

We also considered and selected performance metrics, either for the EDS itself or the entire security system. As a result of our research, we were able to extend our current path evaluation models from those that consider only probability of interruption to a crude, estimated probability of system effectiveness model that could be run on thousands of paths. This should give us a better set of analysis tools to evaluate security systems with EDSs.

Significance

We now have a generic planning process "template" that can be used to create hypothetical planning processes that can then be tested against data to better understand both how attack plans are generated but also where and how to implement preattack security measures.

The adversary-defender model grammar can be used to describe adversary scenarios and defense plans in a precise, unbiased fashion to better evaluate plans used in vulnerability assessments (VAs).

Using the generic planning process, we created an improved method for systematically creating and evaluating plans for multiple adversary attack teams and insiders in collusion with outsider threats. We also extended the existing single-path Estimate of Adversary Interruption computer code that only calculates probability of interruption to the Estimate of Adversary Mission Success (EAMS) computer code that considers preattack factors, interactions between multiple teams, and a simple combat attrition model to estimate the probability that the adversary can complete their mission. We believe that the EAMS computer code can be combined with the multiple team attack methodology to create a better table-top VA methodology.

Finally, all of these approaches can be combined with our engineering understanding of EDSs to provide a systems approach to creating EDSs.

Refereed Communications

M.K. Snell, "Probability of Adversary Mission Success," in *Proceedings of the 47th Annual INMM Meeting*, p. 1, July 2006.

Other Communications

P.B. Merkle, "Extended Defense Systems: I. Adversary-Defender Modeling Grammar for Vulnerability Analysis and Threat Assessment," Sandia Report, SAND2006-1484, Albuquerque, NM, March 2006.

P.B. Merkle, "IED Countermeasures: Vulnerability Analysis and Technology Research," presented at Countering IEDs Europe, Berlin, Germany, May 2006.

P.B. Merkle, "Adversary-Defender Modeling Grammar for Vulnerability Analysis and Threat Assessment," presented at REDTEAM 2006, Albuquerque, NM, March 2006.

Verification and Operation of Adaptive Materials in Space

98294
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Project Purpose

This project builds upon an earlier LDRD project, "Characterization, Performance and Optimization of PVDF as a Piezoelectric Film for Advanced Space Mirror Concepts." This current project focused on building and finalizing three experimental units for testing the materials developed in the earlier project on the International Space Station as part of MISSE-6 (Materials International Space Station Experiment).

This project will expose various piezoelectric polymer films to an actual space environment of atomic oxygen, vacuum ultraviolet (UV), and thermal cycling; we expect to observe synergistic materials degradation. The materials we will evaluate are lightweight, thin piezoelectric polymer films based on polyvinylidene fluoride (PVDF) and its copolymers, which deform in response to controlled charge deposition.

The space exposure experiment, involving various passive and active samples with remote application of excitation voltage and data logging of the materials responses, will provide an avenue to validate the degradation and performance of these materials that was observed in the laboratory environment. The accelerated degradation and performance decrease observed in our laboratory experiments led to our developing a fundamental understanding of the degradation pathways and a preselection of suitable materials.

The actual space exposure will verify these trends and provide feedback on synergistic degradation that is difficult to simulate in a lab setting. This combination of experiments will allow an assessment of the feasibility of using piezoelectric polymer films for advanced low-weight adaptive space mirrors.

FY 2006 Accomplishments

We designed and fabricated three experimental units each with a different experimental focus and delivered them to the National Aeronautics and Space Administration (NASA) at Langley. We provided full details for our experimental package to the NASA launch hazards assessment team.

The first unit contains four passive and four active films samples to be exposed to combined atomic oxygen and vacuum UV. The second unit contains 11 passive and 4 active samples to be exposed only to vacuum UV. The third unit has two passive samples with different thickness of protective aluminum layers to be exposed to atomic oxygen and vacuum UV.

All passive samples will be characterized, both in terms of chemical and physical properties, before and after the mission to determine the effects of the space environmental exposure. Thin foil-type thermocouples attached underneath the samples will measure actual temperatures experienced.

The active experiments were designed to obtain data on the degradation of in situ piezoelectric operational performance of these materials with exposure time. Such piezoelectric data are needed for lifetime and performance predictions of these materials in low earth orbit (LEO) conditions. The active samples are applied in the form of thin film bimorphs designed to move (flex) up and down with applied voltage at a frequency of once per 48 hrs (24 hours up and 24 hrs down). Underneath the tip of each bimorph (i.e., at the point of maximum deflection) is a photodiode sensor measuring the amount of reflected light from the bimorph (reference beam intensity supplied by the photodiode assembly), which with calibrations is used to accurately determine the relative position of the bimorph. The changes in the bimorph position (i.e., how much it will flex up or down with applied actuating voltage) over time are used to determine changes in the piezoelectric responsiveness as a function of LEO exposure.

Measurements of the bimorph position and temperature (simultaneously logged) will be made every 30 minutes, approximately three times every orbit, so that over time data will be available over the entire temperature cycling range (orbit dependent solar position). Various data loggers are used to measure the output from the photodiodes (proportional to bimorph position) and selective temperature locations.

Significance

Our past laboratory-based materials performance experiments have established the major chemical and physical degradation pathways of these materials. The current project leading to the actual space exposure of these piezoelectric polymers will verify the observed trends and provide feedback to assess the feasibility of using piezoelectric polymer films as materials for advanced low-weight adaptive space mirrors.

The project and experiment were described in the *Sandia Lab News* ("Sandia experimental package of piezoelectric films to be part of NASA space station experiment," August 4, 2006.) This will be the first time that PVDF-based adaptive polymer films will be remotely operated and exposed to combined atomic oxygen, solar UV, and temperature variations in an actual space environment. The experiments are designed to be fully autonomous, involving cyclic application of excitation voltages, sensitive film position sensors, and remote data logging. This mission will provide critical feedback on the long-term performance and degradation of such materials, and ultimately the feasibility of large adaptive optical systems using these polymers in space.

A Method to Enable Complex New Software Missions

103394

S. L. Miller, Z. L. Lovelady, S. A. Mulder

Project Purpose

The purpose of this project was to make a preliminary assessment of a newly proposed approach to perform file compression. The assessment entailed two aspects: a) produce a proof-of-concept algorithm demonstrating the basic approach, and b) identify key issues associated with the approach that must be addressed to further assess and implement the basic approach.

FY 2006 Accomplishments

We wrote compression and decompression software that demonstrated two different proof-of-concept algorithms and tested the software using hundreds of files. Our software successfully compressed and decompressed files, with file compression ratios spanning ~ 50 percent to ~ 200 percent, depending on the files. In addition, we identified key issues related to the implementation of the approach.

Significance

The results of this preliminary assessment provide insight into the basic approach and comprise a foundation for further work.

Automated Approach to Dealing with Hacker Attacks

103395

S. Y. Goldsmith, R. B. Smith, M. C. Foehse, S. V. Spires, C. E. Nove

Project Purpose

The primary purpose of this project was to develop a society of intelligent agents that can detect the use of common "hacking" tools on a special-purpose network. Specifically, the agent systems were to learn patterns of use indicative of a group of human hackers operating over the Internet and to identify subsequent situations matching those patterns. The core goal of the project was to develop situation assessment and cooperative data fusion algorithms that enable a group of coordinated agents to identify high-order attack patterns online while they are occurring.

The project is composed of three subtasks: 1) develop a multiagent system for installation on a local network test bed; 2) prepare system components of the test bed with general penetration testing, exploit, and mapping tools obtained through the open Internet; and 3) develop algorithms to train the agents to identify collaborative hacker activities on the network.

- Multiagent system development involves: agent systems requirements and design, agent network platform installation, agent coding and test, and agent system installation.
- Test bed preparation requires the installation of six computer systems on the local test network to host the multiagent system software.
- Agent algorithm development involves creating a situation assessment and mapping algorithm for the monitoring agents and an identification algorithm to classify new instance of situations. Agent training and analysis involves executing an experimental campaign that trains the agents through a series of supervised learning episodes and analyzing the performance of the agents on unseen scenarios.

FY 2006 Accomplishments

Test Bed Installation

- Installation of the network connections for the agent system
- Installation of the five host computers for monitoring agents
- Selection of a candidate set of hacker tools from the Internet
- Installation of a selected subset of hacker tools on the test bed

Multiagent System Design

- Requirements identification for the multiagent system
- Design of the multiagent system architecture and agent components
- Development of the taskable reactive agent
- Development of the monitoring master agent
- Development of the data mining agent

Agent System Algorithm Development

- Statistical analysis of prototypical network data set
- Development of a mapping algorithm for the prototypical network
- Partial development of an identification algorithm

Significance

The results of the project are significant to cyber security systems and processes. Specifically:

- A flexible multiagent system with several innovations that can be applied to other cyber security projects
- A flexible test bed for the study of agent-based cyber systems
- A mapping and data mining facility that can be used to conduct experiments for cyber defense.

Enabling Ultraminiaturization of RF and High-Speed Digital Systems

104420
M. Martinez, D. B. Webb, R. Chanchani, C. A. Apblett, D. Bethke

Project Purpose

High density integrated substrate technology (HDIST) can be used to enable significant size and volume reductions through vertical integration of circuit elements. A suitable technology to enable ultraminiaturization of radio frequency or high-speed digital systems is needed to allow next-generation designs to move forward.

We developed a metal/polymer technology consisting of benzocyclobutene (BCB) separating copper metal layers. We completed and fabricated initial designs, including a five-layer substrate to allow flip-chip attach of a high pin-count microprocessor. As with the development of many (perhaps most) new technologies, the risks associated with the activity are difficult to quantify. As a result, we have not resolved issues associated with the manufacturability and yield of the technology.

We proposed to address manufacturing issues associated with the BCB/Cu technology and demonstrate its potential through a systematic study of defect types and their effects on yield.

The purpose of this project, then, is to:

- evaluate defect densities for the existing process
- identify the most likely defects causing short-circuiting
- correlate visual defects to confirmed electrical defects
- understand which defects drive the low circuit yields.

We will accomplish the visual-electrical correlation with a new, purpose-designed mask set. Once this is accomplished, we will optimize the fabrication process to minimize these defects and assess the feasibility of driving the defect levels low enough to allow actual circuit yield. Assuming the yield is sufficiently improved, we also aim to fully understand the potential application space for this technology.

FY 2006 Accomplishments

- We designed an inspection protocol to collect data on defect types and calculate their densities.
- We tracked defect densities during the process to identify process steps associated with defect formation.
- We fabricated seven substrate lots (six substrates each) through parts of the existing process, with designed variations in key process steps to provoke or suppress suspected sources of defects.
- We determined that airborne particles and fibers do not cause significant defect densities, even when we intentionally used “dirty” processing.
- We found that the density of large defects (e.g., string-like fibers) was significantly reduced by some (largely mechanical) cleaning procedures.
- We found the surface roughness of the starting material (the alumina substrate) sufficient to be partially communicated through to upper layers, despite the planarizing effects of BCB (i.e., the BCB can’t planarize enough to eliminate this initial roughness).
- We found the roughness of deposited (evaporated) metal layers to be severe enough to not be fully planarized by the BCB, and clearly can be bad enough to lead to interlayer shorting if not mitigated, although it is not clear if this is the only defect causing such shorting. Possible causes of this roughness include metal spitting from the source crucible and unoptimized resist profile for lift-off leaving rough edges.
- We saw debris from lift-off (metal stringers, disks, etc.), but the density was far too low to be driving the yield (this was the hypothesized cause of the low yield when the project started)
- We found the density of large defects to be driven primarily by metal patterning steps (i.e., defect density did not change until the next metal patterning step).

Significance

We found “killer” defect (those capable of causing short circuits) density, and therefore yield, to be driven by a small number of factors. These factors do not appear to include general fab cleanliness, or contact lithography-induced defects (e.g., photoresist being pulled off wafer by mask, leaving a void), which was the initial hypothesis. While it is still possible that such defects are important, and occur with such a low density that a much larger statistical sampling is required to show this, the data collected to date does not show this.

Instead, the data indicates that some combination of substrate roughness, metal roughness, or lift-off debris is responsible. The apparent mechanism is that these things produce defects that are too high for the BCB to planarize, so they poke through, providing a connection between conducting layers (i.e., a short circuit).

These are significant findings, as they indicate that the causes can be addressed to improve yield significantly. The approaches might include using substrates that are smoother from the start (e.g., polished GaAs wafers), reducing metal deposition rate to eliminate spitting, modifying other metal deposition parameters to optimize smoothness, optimizing the metal lithography to enhance the profile for easy lift-off, using an automated lift-off tool that filters out lift-off debris, and other, similar approaches.

We showed that the yield drivers can be understood and controlled. Without work directly addressing the causes of low yield, it is impossible to state whether yields can be improved enough to produce functioning large-area die with a reasonable number of substrates processed. At this time, though, it does seem likely that this can be achieved.

Thermo-Optic Focal Plane Array (TO-FPA) for High Sensitivity Room Temperature Infrared Imaging

104421

M. Watts and G. N. Nielson

Project Purpose

This project was established to investigate the potential of a thermo-optic plane array (TO-FPA) for room-temperature infrared imaging. Specifically, the goals were to firmly establish the theoretical limits of the TO-FPA approach, develop designs for TO-FPA sensor implementations, and evaluate these designs in terms of noise performance, power consumption, readout techniques, and the fabrication processes required to implement these approaches.

Current infrared imaging focal plane arrays such as those based on mercury cadmium telluride (MCT), type-II detectors, and bolometers require substantial cooling to approach background limited performance (BLIP). However, cooling MCT arrays consumes considerable power and typically requires a cryostat. In the case of a soldier, cooling detector arrays requires transporting large numbers of batteries. A typical set of infrared binoculars consumes a pair of D-cell lithium batteries every four hours. In satellite and unmanned aerial vehicle applications, eliminating the need for cryogenic cooling of infrared sensors will enable a significant reduction in payload weight (up to ~ 20-30 percent) and power consumption.

In nearly all of these applications, the utility of the thermal imager is determined by its sensitivity, expressed in terms of noise equivalent temperature difference, noise equivalent power, or detectivity; the more sensitive the thermal imager, the greater the standoff distance.

The TO-FPA approach uses the highly sensitive thermo-optic effect in integrated optic microring-resonators coupled to infrared absorbing elements to detect minute changes in the microring-resonator temperature caused by the absorbed infrared radiation. Initial calculations indicate that the technique's massive scale factor (~ 25,000 X larger than bolometers), passive operation, and absence of metal thermal paths should enable near BLIP in a room-temperature infrared imaging system.

Recent advances in microphotonic circuits open up the possibility for integrating large arrays of microring-resonator elements and the potential for a new more sensitive method for room-temperature infrared imaging. In addition, the TO-FPA approach uses microelectromechanical systems (MEMS)-style complementary metal-oxide semiconductor (CMOS) compatible processing, fabrication techniques that have been well developed in Sandia's Microsystems and Engineering Sciences Applications (MESA) complex and offer the potential for direct integration with CMOS circuitry.

The purpose of this "feasibility" project is to:

- consider the impact that recent advances in microphotonics offer for improved room-temperature infrared imaging
- consider the fundamental limits of thermo-optic infrared imaging
- develop realistic CMOS compatible TO-FPA designs
- generate and use finite element models of these designs to establish thermal properties of the sensing elements
- address system issues such as power consumption and readout techniques
- develop and evaluate approaches for fabricating these sensors.

TO-FPA has the potential to enable a substantial improvement in sensitivity over all existing room-temperature infrared imagers, can be passively remoted, and should use substantially less power than approaches requiring cryogenic cooling.

FY 2006 Accomplishments

We investigated the theoretical limits of the TO-FPA approach, established candidate sensor designs, developed finite-element models, and investigated fabrication techniques. Our theoretical investigations took a look at the impact of thermal phonon noise, readout shot noise, and shot noise caused by uncooled telescope walls. Importantly, we determined that there are no inherent limitations to reaching the thermal phonon noise limit with this TO-FPA technique. This is in contrast to conventional bolometric techniques, which suffer from readout shot noise and 1/f noise and are typically limited to sensitivities that are one to two orders of magnitude above their phonon noise limits.

Because the TO-FPA requires only dielectric connections (i.e., no metallic wires), it should be possible to reduce the thermal phonon noise limit relative to what is achieved with conventional bolometric techniques. To bolster this argument, we developed finite element models of representative TO-FPA sensor designs. From these models we were able to establish the ability to achieve thermal conductivities of $\sim 10^{-8}$ W/K, which is an order of magnitude below what has been demonstrated in conventional bolo-meters. With thermal conductivities this low, the phonon noise limit in the TO-FPA should reach a NEP = $2.4\text{x}10^{-13}$ W/(Hz$^{1/2}$) (i.e., D* = $1.6\text{x}10^{10}$ cmHz$^{1/2}$/W at a 40 µm pixel size).

We developed two candidate sensor designs, one that uses a silicon-based sensing element and one that uses a silicon nitride-based sensing element. We are pursing two approaches because there are advantages and disadvantages to each and it is not clear which approach will win out. Both designs are currently being scheduled for fabrication in Sandia's MESA complex.

The silicon approach is based on a silicon-on-insulator process that takes advantage of the relative ease in which silicon can be selectively oxidized. Selective oxidation allows for a silica scaffold to be directly incorporated into the design to enable very low thermal conductivity. Silicon also has the advantage of being able to tightly confine an optical mode. This allows for very small microring diameters (< 5 microns) and, thus, pixel sizes. However, since silicon does not absorb in the infrared, a separate absorbing layer will be required. Silicon nitride is a strong absorber in the 8-12 µm infrared regime. As such, the microring (or microdisk) resonator can serve both as the optical and absorbing elements.

In summary, we established the fundamental limits governing the performance of TO-FPA-based infrared sensors, developed candidate sensor element designs (one in silicon and one in silicon nitride), established the thermal properties of each using finite element analysis, and established paths for fabricating each design in Sandia's MESA complex. The analysis confirmed initial indications that reaching the phonon noise floor should be possible with TO-FPA technique. Although much effort is still required to complete the TO-FPA designs and develop fabrication processes, our candidate designs appear to lend themselves to established CMOS fabrication techniques.

Significance

We have demonstrated that the TO-FPA approach has the potential to reach the thermal phonon noise limit, a feat that has not yet been demonstrated with bolometric or any other thermal detectors. Moreover, we demonstrated that the TO-FPA sensing elements can be designed to have a lower phonon noise limit than bolometric techniques. In addition, we developed a pair of CMOS compatible sensor designs that we believe can be fabricated with MEMS-style CMOS processing. We also investigated system-level issues such as arrayed configurations, readout approaches, and power consumption.

At all levels the TO-FPA approach exhibits promise to approach background limited performance. If this is in fact possible, the TO-FPA will open up a new era of room-temperature infrared imagers. The TO-FPA will enable uncooled imaging systems to replace existing cooled approaches such as MCT detectors in many systems where weight and power consumption are critically important.

Enhancement of HPM Effects

104480
L. D. Bacon

Project Purpose

The paradigm of high-power microwaves (HPM) is shifting from that of large pulsed power machines to smaller systems that operate with a high repetition rate. In the newer systems, higher powers are generated by ganging systems together in parallel. The payoff is the ability to address a new set of targets, including improvised explosives devices and their offspring, with small, versatile, user-friendly systems.

The critical issues for this new technology are the repetitive operation (rep rate), synchronization of multiple modules, and understanding/optimization of the effects. Our main goal is to demonstrate the effectiveness of very high repetition rate pulses against the electronics of targets of interest.

FY 2006 Accomplishments

Our work added to the database of radio frequency/HPM effects on electronics at high repetition rates. We observed significant lowering of effects thresholds for some targets of interest. The experimental effects work was carried out in our gigahertz transient electromagnetic (GTEM) test chamber.

We also identified candidate charging and switching topologies potentially capable of achieving these rates. They include a transformer-based system with solid-state switching in the primary, blocking oscillator and autotransformer topologies, and compact pulse-forming-lines with spark-gap and photoconductive semiconductor switches.

Optical triggering has large payoffs in switch placement within the HPM source/antenna and in multielement array applications, where each element can operate at low power, and high power densities are created by combining the radiated fields in space. This can lead to a further advantage of electronic beam steering.

Significance

Making HPM systems more effective will allow a reduction in their size and power requirements. Reduction in size has great implications for Department of Defense and Department of Homeland Security, enabling weaponizable systems that may negate the use of electronic systems, including improvised explosive devices. Also, the understanding of high repetition rate effects will increase our understanding of the susceptibility of DOE assets.

Updating Time-to-Failure Distributions Based on Field Observations and Sensor Data

104511
D. Briand, D. N. Shirah, K. S. Lowder

Project Purpose

One of the primary purposes of our modeling efforts is to use component/system reliability estimates along with inventory levels, maintenance and inspection schedules, and operational requirements to optimize supply/repair chain processes. Typically, we model relatively complex systems or system-of-systems with hundreds to thousands of components. Many types of components typically will have a bathtub-shaped failure rate life distribution. This distribution is characterized by a decreasing failure rate during the early ("infant mortality") portion of its life, a constant failure rate during the useful (random failure) portion of its life, and an increasing failure rate during the rapidly aging (wear-out) portion of its life.

During the infant mortality phase, failures are typically caused by manufacturing defects. During the random failure phase, component failures are usually caused by chance, perhaps as a result of overstress or a shock to the system. The wear-out phase of a component's life is characterized by wear or accumulated damage that exceeds allowable limits for normal operation. In many models and analyses, the failure rate distributions for components model only the random failure period, typically with a constant failure rate. However, when optimizing supply/repair chain processes in a large-scale logistics support model, being able to use the failure characteristics across a component's lifetime provides greater accuracy and usefulness of the model.

To help simplify the component failure characterization process, Sandia has developed and is using a bathtub-shaped combined lifecycle (CMBL) time-to-failure (TTF) distribution that represents the entire infant mortality, random failure, and wear-out phases of a component's lifetime with parameters that make it relatively easy to elicit the probability of failure distribution from subject matter experts and available data. The five parameters are 1) the fraction of failures expected during infant mortality, 2) the duration of the infant mortality period, 3) the fraction of failures during the random failure period, 4) the mean of the wear-out period, and 5) the standard deviation of the wear-out period.

For this CMBL distribution and other sectional distributions, the biggest gap in realistic component lifetime estimation is developing theoretically valid methods for updating combined TTF distributions as lifetime hours are accumulated. In other words, as parts undergo the infant mortality, random, and wear-out phases of their lives, how should the uncertainty in the components' lifecycle distribution be adjusted to reflect real-world failure behavior?

The objective of this project was to address this gap in component lifetime estimation. More specifically, the goal was to understand how to update the CMBL and other CMBL TTF distributions given new field observations and sensor data, and to investigate/develop the theoretical methodology that supports this process. To make optimal operational/maintenance decisions based on updated TTF distributions, we must be able to analyze the accuracy, precision, and confidence inherent in those distributions.

FY 2006 Accomplishments

We investigated two different approaches to updating combined lifecycle TTF distributions. The first approach updated each section, infant mortality, random, and wear-out phases separately. This approach depended upon the iterative process within the CMBL distribution to guarantee continuity to help provide convergence from the distribution represented by the input parameters to the distribution of the new data.

The second approach we used, a Bayesian updating methodology, assumed the distribution for the new data (time of new failure) was the same as the distribution where the new data occurred in the component's lifecycle, i.e., if the new data occurred in the random failure section of the component's lifecycle, the new data was assumed to have an exponential distribution with a constant failure rate. A conjugate prior distribution for each section was also used since it provided a well behaved closed form solution and has well documented use for reliability implementation. From the resulting posterior distribution, the updated parameters of the combined TTF distribution were either directly obtained or derived.

To help facilitate the evaluation of the updating process of the first approach, a stand-alone Visual Basic routine was created to calculate the combined lifecycle distribution, provide the critical parameters that defined the distribution in addition to the input parameters, and populate a data file that contained the critical parameters and data values for demonstrating convergence to specified distributions. Also, the routine automatically calculated and graphed the limits of the input parameters – necessary for a complete parametric evaluation of the distribution.

The results of this approach were positive but incomplete. Convergence to the distribution of the data occurred relatively quickly when the mean of the data varied from the original combined TTF distribution. However, when only the fraction of random failures was changed, convergence to the distribution of the data did not occur but remained essentially unchanged. The problem seems to lie with the fact that the random section of the distribution is truncated exponentially, yet the Bayesian updating process is referencing a nontruncated exponential.

The second approach relied on the development of a closed-form solution to a slightly modified version of the combined lifecycle distribution. The closed-form input parameters were 1) the fraction of failures expected during infant mortality, 2) the failure rate of the infant mortality portion, 3) the failure rate for the random failure portion, 4) the mean of the wear-out portion, and 5) the standard deviation of the wear-out portion. The updating methodology attempted required a method for weighting the expert's estimate of the parameters and then a recalculation of the closed-form solution for each additional new data (failure) experienced.

The results of this second approach appear very promising given that the derivation of the closed-form solution is theoretically valid. Also, the fact that a large number of data points is needed in order to establish a good approximation for the distribution, combined with the reality that such extensive failure data is seldom available, makes a strong argument that an alternative approach to updating the parameters should be investigated.

Significance

The results obtained in evaluating these methods for updating TTF distributions show promise although more research is needed. The methods developed in this effort for updating the CMBL and other TTF distributions should be valuable in enhancing maintenance planning and real-time situational awareness processes.

These methods, used in enterprise level and prognostics and health management (PHM) modeling, should:

- help provide more accurate instant feedback on the current status of equipment
- provide tactical assessment of the readiness of equipment
- identify parts, services, and so on that are likely to be required
- provide a realistic basis for scheduling and optimizing equipment maintenance schedules
- help ensure that the useful life of expensive components is taken full advantage of while reducing the incidence of unplanned maintenance.

One recommended approach to using these updating schemes would be to run the updating process independent of the enterprise level or prognostic model. Since the updating process could take considerable time depending upon the number of components being modeled, a preprocessor would take the updated data from field databases and, where applicable, update each component's TTF distribution prior to enterprise level or PHM model runs. In this way, the data updating process does not interfere with the run times of the large-scale enterprise level or PHM models.

This approach to using these updating schemes could benefit the Sandia/Lockheed Martin-developed Support Enterprise Model for use in the Joint Strike Fighter program, and the Sandia-developed Real Time Consequence Engine for use in the Missile Defense Agency's Airborne Laser program.

Some of these techniques should apply to other sectional distributions as well. In particular, the updating process that treats each section of the bathtub distribution independently should apply to any sectional distribution that uses an iterative scheme for determining the boundaries between the three sections of a component's lifecycle. The approach taken to develop the closed-form distribution should provide a universal model for most reliability-based TTF distributions.

Additional research is necessary to investigate some of the identified drawbacks and to determine if updating either the combined lifecycle distribution or the closed-form solution with sets of new data (instead of one data point at a time) will provide the same or perhaps better/quicker results, thus broadening its applicability.

Refereed Communications

D. Briand, J.E. Campbell, and A.V. Huzurbazar, "Updating a User-Friendly Combined Lifetime Failure Distribution," to be published in *Proceedings of the Annual Reliability and Maintainability Symposium (RAMS)*.

Radical Advancement in Multispectral Imaging for Autonomous Vehicles (UAVs, UGVs, and UUVs) Using Active Compensation

104514

D. V. Wick and B. Bagwell

Project Purpose

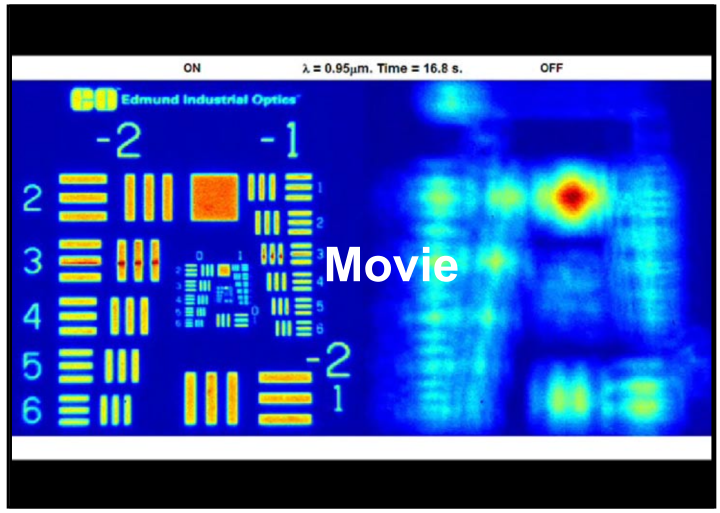
Axial (or longitudinal) chromatic aberration is a variation in focal length *f(λ)* with wavelength. In addition to the spectral response of the focal plane array/film, it is often the limiting factor determining the usable spectral bandwidth of a refractive (i.e., transmissive) imaging system, especially near the optical axis. In the case of a single positive lens, shorter wavelengths (e.g., blue) will have a shorter focal length than longer wavelengths (e.g., red). Further image degradation associated with dispersion results from transverse chromatic aberrations and the wavelength dependence of the so-called monochromatic aberrations, especially at large field angles.

Traditionally, more lenses with different glass types are added to the optical system in order to increase the spectral bandwidth over which the system meets some required metric (e.g., diffraction/detector limited, minimum spatial resolution, and so on). Mechanical color wheels can also be used in tandem with an adjustable focus if spectral discrimination is desired, typically over a narrow field of view.

The purpose of this project was to develop and demonstrate an alternate solution by using a liquid crystal (LC)-based polarization interference filter to isolate a narrow spectral region of interest and LC lenses to correct the aberration (defocus) over that spectral region. The end state would be a transmissive system that is compact, requires no macro moving parts, and has the ability to spectrally resolve an image.

FY 2006 Accomplishments

We demonstrated a compact, multispectral, refractive imaging system that has an increased operational bandwidth, provides for spectral selectivity, and nonmechanically corrects aberrations induced by the wavelength-dependent properties of a passive refractive optical element (i.e., lens) in the system. We submitted a patent application on the concept and identified several applications where this technique could be useful. Specifically, we identified night vision systems and wide-band multispectral systems as two areas where this technique would provide improved imaging capabilities. We are currently working with potential customers to further develop these ideas.



This shows images taken with a simple imaging system using a polarization intereference filter and liquid crystal lens. The right shows the image without correction, but the left shows the image when chromatic aberrations are corrected in real-time.

Significance

We identified night vision systems and wide-band multispectral systems as two areas where this technique would provide improved imaging capabilities. Autonomous vehicles have become multimillion-dollar industries supplying both commercial and military customers. As evidenced by recent events in Iraq and Afghanistan, there is a growing sense of urgency to improve our ability to discriminate spectral content for a number of military applications, including chemical/biological detection and the detection of improvised explosives devices.

As size, weight, and power are a premium on many platforms, especially on mini and micro unmanned aerial vehicles, this technique can reduce the size of the optical system and allow for spectral resolution from a compact, nonmechanical system. It is also possible to use this technology in conjunction with nonmechanical zoom, which is also being developed by our group at Sandia. The combination of increased spectral and spatial resolution could be extremely useful for threat discrimination from an extremely compact, nonmechanical system.

In addition, night vision systems are currently limited to a spectral bandwidth of approximately 600 nm to 850 nm. Recent Defense Advanced Research Projects Agency-funded research has led to the development of an InGaAs-based detector with a very wide spectral range, spanning the visible (0.4-0.75 µm), near-infrared (NIR: 0.75-1.0 µm), and short wave infrared (SWIR: 1.0-1.8 µm). There are at least two companies that have developed complex imaging sensors that will image across all of these bands. Our technique would not only make the system more compact, it would also allow spectral discrimination from a nonmechanical system.

Other Communications

B.E. Bagwell, D.V. Wick, R. Batchko, J.D. Mansell, T. Martinez, S.R. Restaino, D.M. Payne, J. Harriman, S. Serati, G. Sharp, and J. Schwiegerling, "Liquid Crystal-Based Active Optics," in *Proceedings of the SPIE Optics & Photonics*, August 2006.

Biodegradation of Hermetic Seals

104574
R. P. Toth, D. D. Burchard, D. A. Summers, A. Allen

Project Purpose

The purpose of this work was to develop faster and more sensitive methods for leak detection of glass-to-metal hermetic seals and to investigate the impact, as well as mechanisms, of environmental factors that could affect the seals' performance.

FY 2006 Accomplishments

We used Kovar-glass seals in a TO-3-type power transistor for our test materials. We devised a test method based on a modification of helium (He) leak testing. Rather than prolonged exposure to He and then measuring the egress of He, we filled the can with He via a portal, then measured He leakage around the base and emitter leads with a mass spectrometer. We evaluated environmental factors with humid atmospheres and common soil samples and monitored the parts over the remainder the project period.

Significance

The significance of our work is two fold: 1) the development of a rapid and more sensitive method of testing glass-hermetic seals in a power transistor test bed; and 2) evaluation of environmental factors that affect glass hermetic seals in this test bed for a period of less than six months.

Optimized Custom Knowledge Discovery

104577

G. N. Conrad, P. Zhang, S. M. Eaton, C. M. Wray

Project Purpose

The purpose of this project is to develop and demonstrate how knowledge discovery (KD) technologies can be employed and modified to address national security needs.

FY 2006 Accomplishments

We adapted existing KD technologies to provide a demonstrable capability for a national security need. Specifically, we used a support vector machine (SVM) algorithm for text classification. SVMs use a machine-learning approach that produces a model from exemplary data. The models are then used to classify new, unknown text documents. This approach allows large bodies of textual files to be searched for specific content rather than the less efficient approach of searching the repositories for keywords. To accomplish this we explored methods for establishing a set of content domain specific terms that is used by the SVM learning and classification processes. These content domain specific term lists form the foundation not only for our SVM algorithms but also form the basis of domain specific ontologies that can be used by other KD algorithms.

Significance

The methods explored and developed in this project will improve the capabilities and efficiencies of knowledge workers who deal with large repositories of national security data.

Active Resonant Subwavelength Grating for Scannerless Range Imaging Sensors

104889

S. A. Kemme, D. W. Peters, T. Carter, R. O. Nellums, A. A. Cruz-Cabrera

Project Purpose

In this project, we designed and obtained initial results for a wavelength-agile, high-speed modulator that enables a long-term vision for the terahertz scannerless range imaging (SRI) sensor.

This new component would take the place of the SRI microchannel plate, which is limited to photocathode sensitive wavelengths (primarily in the visible and near-infrared regimes). The new component is an active resonant subwavelength grating (RSG). An RSG functions as an extremely narrow wavelength and angular band reflector, or mode selector. Theoretical studies predict that the infinite, laterally extended RSG can reflect 100 percent of the resonant light while transmitting the balance of the other wavelengths. Experimental realization of these remarkable predictions has been hampered primarily by fabrication challenges. Even so, we demonstrated large-area (1.0 mm) passive RSG reflectivity as high as 100.2 percent, normalized to deposited gold. In this work, we transform the passive RSG design into an active laser-line reflector.

We accomplished the two main milestones of this three-month project: 1) numerically characterize the wavelength and angular bandwidths of a candidate RSG design as well as the overall efficiency, and 2) identify and characterize a candidate material for the variable index layer that will efficiently modulate the RSG.

FY 2006 Accomplishments

We investigated various methods of creating an active RSG. We decided to use a material with an index of refraction that we can vary with voltage: an electro-optic material. We then considered which materials and configurations we could use in such a device as well as the electrode geometry.

Linear electro-optic materials allowed us to vary the refractive index with the application of an electric field. We considered materials that exhibit a strong electro-optic effect, have low-loss in the wavelength range of interest, and are compatible with the fabrication steps required for the RSG process. Candidate materials are lithium niobate, lithium tantalite, potassium titanyl phosphate (KTP), barium metaborate, barium titanate ($BaTiO_3$), and lead lanthanum zirconium titanate (PLZT).

Possible electrode geometries for applying the voltage are frames, interdigitated electrode fingers, or a combination. Frames offer the advantage of keeping the optical transmission portion of the device free of all optically absorbing electrical regions, maximizing the optical performance. This approach requires high voltages to obtain the field strength required as a result of the noncentralized location of the electrodes. Interdigitated electrodes inside the optical area of the device can reduce by orders of magnitude the distance between electrodes and thus reduce the required electric field by a similar amount. Interdigitated electrodes will interfere with the optical properties due to their location in the optical path.

We numerically explored various geometries of $BaTiO_3$ guiding layers on a silicon dioxide substrate. We employed grating materials consisting of silicon dioxide, $BaTiO_3$, indium tin oxide (ITO), and combinations of these. The higher-index $BaTiO_3$ creates a wider wavelength resonant peak than the lower-index silicon dioxide. ITO offers a conductive, mostly transparent material for electrical connections with active control of the device response. However, ITO is lossy, which can detrimentally affect device performance.

We also numerically modeled designs that incorporate ITO electrodes over a layer of $BaTiO_3$ on a silicon dioxide substrate. This combination proved to offer the most promise for an active device using interdigitated electrodes without significant loss. The high-index $BaTiO_3$ leads to a tightly confined mode. Placing a grating of ITO directly on the $BaTiO_3$ guiding layer leads to absorption loss. Dividing the grating into two parts, with a lower buffer layer of $BaTiO_3$ topped with a layer of ITO for an electrode, allows the lossy ITO to be separated from the high electric fields of the guiding region, thus minimizing loss.

A design consisting of 200 nm of uniform $BaTiO_3$ for the guiding layer and a 425 nm grating consisting of 180 nm of $BaTiO_3$ and 120 nm of ITO leads to a low-loss alternative. The device has electrical contacts in the active area while minimally affecting performance. Rigorous coupled wave analysis calculations indicate a peak reflectivity for this configuration of 99.5 percent, indicating that loss from the ITO is minimized. The transmission minimum is at the designed 850 nm and has a full width at half maximum (FWHM) of 20 nm.

Significance

The active RSG acts as a wavelength-agile, high-speed modulator that will enable a long-term vision for the terahertz SRI sensor. The active RSG takes the place of the currently used SRI microchannel plate, which is limited to photocathode sensitive wavelengths (primarily in the visible and near-infrared regimes).

Two of Sandia's successful technologies – subwavelength diffractive optics and terahertz sources and detectors – are poised to extend the capabilities of the SRI sensor. The long-term goal is to drastically broaden the SRI's sensing waveband – all the way to the terahertz regime – so the sensor system can see through image-obscuring, scattering environments like smoke and dust. Surface properties, such as reflectivity, emissivity, and scattering roughness vary greatly with the illuminating wavelength. Thus, objects that are difficult to image at near-infrared wavelengths may be imaged more easily at the considerably longer terahertz wavelengths (0.1 to 1 mm).

Sandia invested considerable effort on a passive RSG two years ago, which resulted in a highly efficient (reflectivity greater than gold), wavelength-specific reflector. In this project, we leveraged this passive RSG work for extension to the active RSG concept.

SRI sensing is a flagship technology that Sandia developed and has applied to various projects, from underwater imaging in turbidity for the Navy's Coastal Systems Station to structural dynamics measurements and space vehicle inspection for National Aeronautics and Space Administration's international space station. Sandia has thrived with each SRI sensor delivery, and every field installment benefits from a multitude of engineering evolutionary improvements. However, to continue on this successful path, Sandia must take the opportunity to develop fundamental, disruptive technologies and apply them to systems such as the SRI sensors. This RSG demonstration is key to significantly advancing the SRI sensor family.

Development of Nanofluidic Devices for Dielectrophoretic Chromatographic Separation of Biomolecules

104975
A. J. Skulan, S. Pennathur, B. A. Simmons, S. S. Mani

Project Purpose

The purpose of the project is to develop and validate a new nanofluidic (100-1000 nm length scale) separation technique that exploits selective transport of biomolecules based on their mobilization characteristics in applied nonlinear electric fields. This approach will allow the separation of biomolecules such as proteins and DNA and may become an enabling technology in the isolation and identification of biomarkers in disease response.

Biomolecule-selective transport in the proposed devices is postulated to occur when nonlinear electric fields at the interface of two geometrical regions in an electrokinetic flow are coherently reinforced by regularly patterned arrays of structures such as ridges or posts. We will follow an iterative approach of simulation, device design, manufacture, experimental testing, and results analysis. The presence of each of these steps and their underlying enablers – physics modeling and simulation, high-precision fabrication, and the unique perspective of our university collaborators – greatly enhances the quality of the research performed while accelerating the progress of the research.

We anticipate that the underlying nonlinear separations mechanism will produce capabilities superior to conventional biomolecular chromatography. At a minimum, it will enable a confirmatory separations technique that is complementary to conventional methods while possessing improved characteristics in other performance aspects. In addition, this technique may enable us to perform separations of biomolecules that currently cannot be performed in free solution, such as DNA separation and chiral molecule separation.

The chromatographic capabilities promised by these devices are of fundamental interest to the Department of Energy (Office of Science, Division of Chemical Sciences, Geosciences, and Biosciences). Bioagent and biotoxin detection and analysis employing these new devices is of practical interest to the Department of Homeland Security and the Department of Defense by virtue of their potential sensitivity, operational simplicity, mobility, and cost effectiveness.

FY 2006 Accomplishments

We attained a number of key accomplishments in the areas of design, fabrication, and operation.

Design: We implemented the faceted prism design methodology we previously developed in the design of composite microfluidic-nanofluidic devices. We generated seven different channel geometries in this process, including either single channel or concatenated multifacet designs with a variety of interface geometries. Some channels included crossed injector ports to permit the injection of narrow analyte bands.

Fabrication: We combined these designs into a series of circular 4” wafer masks that permitted mass replication of the seven channel designs, with channel redundancy (14 channels total per wafer). We fabricated these in glass using anisotropic plasma etching methods, resulting in devices with sharp micro/nanochannel interfaces. The successful execution of this difficult fabrication is a key accomplishment and adds a highly leverageable capability in composite micro/nanofluidics to Sandia.

Operation: We tested the operation of this suite of devices using a variety of analytes that included both organic small-molecule fluorescent tracers (e.g., fluorescein) and fluorescently tagged proteins. One such protein was Alexa Fluor-488 tagged C-reactive protein, a key biomarker of acute inflammation that is diagnostic of a wide range of inflammatory maladies including: bacterial, viral and fungal infections, cancer, and pneumonia. We demonstrated ~ 1000-fold concentration of this protein in 1 mM buffer at 100 V in 50 sec, while no concentration was observed under the same conditions with 100 mM buffer. Initial simulations suggest that this concentration behavior arises due to an increase in the Debye layer thickness with decreasing ionic concentration in the channel.

The second approach we investigated was to take the insulating-post array design paradigm that is currently being successfully applied to microfluidic insulating dielectrophoresis (iDEP) and shrink it from the microscale to the nanoscale. This constituted a fabrication challenge, as the fabrication was performed using polymer injection molding techniques. This had not been achieved previously and would constitute a significant improvement over current state of the art performance. We replicated the nanoscale post-shaped features following process optimization and successfully bonded them to produce nanofluidic devices.

Significance

The results of this initial cursory investigation into the use of nanofluidic devices for the concentration and separation of proteins are applicable to a wide range of problems in Sandia's homeland security, energy, and military applications mission areas. The advances most useful to future efforts are in manufacturing process experience and protein concentration for protein-related projects.

Manufacturing: We successfully introduced the design, fabrication, and operation of nanofluidic devices to the list of Sandia's areas of expertise with two separate approaches – anisotropic plasma etching of hard substrates (demonstrated for quartz) and injection molding of polymers (demonstrated for Zeonor-1060). Each approach presented process challenges at the nanoscale that were solved through experimentation and consultation with collaborators. These capabilities can be applied to research activities requiring the transport of picoliter volumes of fluids, including the analysis of proteins from individual cells and bacteria for homeland security, defense, and water quality assurance applications.

Protein concentration: The ~ 1000-fold concentration of proteins at a depthwise interface has wide application to homeland security, defense, and water quality assurance problems where the native protein concentration is below the detection limit. This is often the case as analytes can be in the parts per billion or parts per trillion range for these applications. Concentrating analytes on-device without requiring external reagents or consumable filter cartridges represents a significant advantage for stand-alone or continuous monitoring applications.

Advanced Robot Locomotion

105067

R. H. Byrne, J. C. Neely, D. K. Novick, B. R. Sturgis, D. G. Wilson, S. E. Rose, S. Buerger

Project Purpose

The purpose of this project is to build on Sandia's expertise in energy systems and control systems for mobile platforms to outline the development of a hydraulic-powered walking robot. By focusing on the mobility area, this project differs from other humanoid efforts and focuses on the areas that would have the highest payoff for Sandia and our DOE, military, and intelligence customers. The main milestones for this effort are a dynamics test bed for walking, models for analyzing different control strategies, and preliminary control algorithms for walking.

FY 2006 Accomplishments

We developed several dynamics models for evaluating different walking control strategies. We also developed a dynamics test bed that consists of a double inverted pendulum for demonstrating different control system strategies. In addition, we developed several optimization and control strategies that were evaluated through simulation.

Significance

This effort has increased our technical capabilities in modeling, simulation, and control of walking locomotion systems.

Enabling Technology for Shape Optimization of Armor and Other Defense Assets

105127

S. J. Owen, T. J. Tautges, S. L. Brown

Project Purpose

The purpose of this project was to integrate the DDRIV shape optimization geometry and mesh support tool into the DAKOTA optimization toolkit and use the tool to perform shape optimization of armor designs.

FY 2006 Accomplishments

We integrated DDRIV into the DAKOTA optimization toolkit, and wrote documentation for the DDRIV tool and library. We developed preliminary geometric models for a radio frequency switch MEMS (microelectromechanical system) design to test the system, but did not perform shape optimization of this component.

Significance

Integration of DDRIV shape optimization capabilities into the DAKOTA optimization toolkit may allow shape optimization of other designs in the future. Documentation of DDRIV tool options will be useful to the general scientific community, particularly the DOE Scientific Discovery through Advanced Computing (SciDAC) community.

Discrimination of Signatures for the Modern Battlefield

105675

M. J. Kaneshige, L. A. Boye, M. M. Hoffman

Project Purpose

Novel and advanced energetic materials are in development around the world. The purpose of this project was to explore the possibility that these materials can be discriminated based on optical or other signatures.

Stand-off detection of unique signatures from explosive events could be used to identify battlefield threats or to track development efforts by potential adversaries. Extensive data have been collected from a variety of explosive events, involving both conventional and enhanced-blast explosives, and these data are available for evaluation of spectral patterns related to ingredients, geometry, size, and so on. In addition, predictive methods exist for estimating optical signatures (spatial, spectral, and temporal) from various explosives.

These analytical methods can be used to extend the experimental database beyond the experimental parameter space, and to suggest measurements with high sensitivity to the explosive characteristics of interest. For instance, certain ingredients may be most easily identified through detection in certain wavelength bands (individually or in combination), but this must be considered in the context of atmospheric absorption. Ultimately, a thorough evaluation of the characteristic signatures of different explosives, whether experimental or computational, can be used to evaluate data collected by existing sensor systems, and to guide development of new sensor systems to optimize their suitability for discrimination.

FY 2006 Accomplishments

We consolidated and organized spectral data previously collected at several wavelengths during explosive tests performed at Sandia in preparation for insertion into an existing database of explosive spectral data.

We identified a computational fluid dynamics code with appropriate chemistry, physics, spectral emission, and atmospheric transmission models. We will use this code to compare with our collection of data and possibly enhance or extend conclusions drawn from them.

We met with representatives of Applied Research Associates, CRAFT Tech, and various Sandia organizations to evaluate the state of the art and develop a path for future work.

Significance

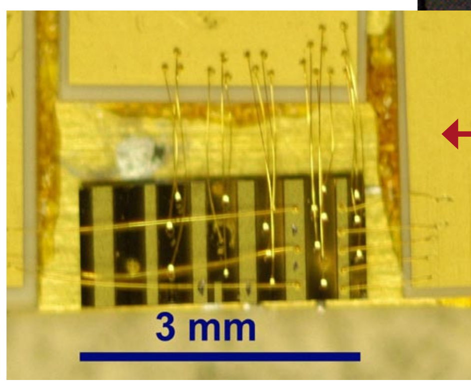
This work will contribute to several Sandia mission areas, including intelligence assessments, science and technology, and nonproliferation. We collaborated with other groups working in these areas with a variety of technical and programmatic backgrounds, both inside and outside Sandia, and this project therefore served to increase the impact of the individual groups and Sandia as a whole. For instance, the enhanced blast explosive test capabilities at Sandia generally are focused on the science and technology of blast performance, but we have been able to collect spectral data at negligible cost, increasing the relevance and reducing the relative cost of the experiments.

Adding these data to an existing database of similar data from a large number and variety of historical explosive events extends the usefulness of the database, not simply by summation, but by providing a basis for comparison between a growing number of signatures. Indeed, the science and technology goals of the explosive testing program include the acquisition of a number of other measurements (pressure, imaging, and so on) that have the potential to provide greater physical insight into the signatures, which otherwise may invite speculation regarding interpretation of differences and anomalies. Tight control and characterization of the experimental parameters makes the resulting data ideal for basing assessments. In turn, the spectral data have the potential to provide physical insight of a different nature than the conventional blast diagnostics.

In other words, one significant aspect of this work is the leverage gained by bringing together several otherwise distinct efforts. A significant result is the development of a consortium of research and development organizations, with Sandia in the lead, to develop the existing database and combine it with predictive simulation capabilities to create a new science-based discrimination capability for US intelligence organizations.

Grand Challenges

Replace This:
- Molecular gas FIR laser
- Size: ~ 1 m x 0.3 m, 25-75 kg
- Fragile precision alignment
- One wavelength at a time
- 40 mW at 2.5 THz



With This:
- Semiconductor THz QC Laser
- Size ~ 2 mm x 0.1 mm, 1 g
- No mechanical parts
- Scalable to mass production
- Multi-wavelengths straightforward
- 50 mW at 3 THz, 250 mW at 4 THz

Six QCLs integrated on one chip

95214, Terahertz Microelectronic Transceiver (TμT) System

Grand Challenges are audacious, high-risk, high-value, multidisciplinary, multimillion-dollar projects that target some of the most difficult science and technology problems that could impact national security. These projects pursue groundbreaking research and development that could affect or revolutionize science and technology at the system level.

A successful Grand Challenge seeks to create significant science and technology advances that create forefront capabilities and provide multiple long-term benefits across national security mission areas. It also seeks solutions to critical problems, such as making pulsed power an attractive path to fusion energy; developing innovative solutions to national security space monitoring; and creating and using analytical tools that can improve the early diagnosis of infection.

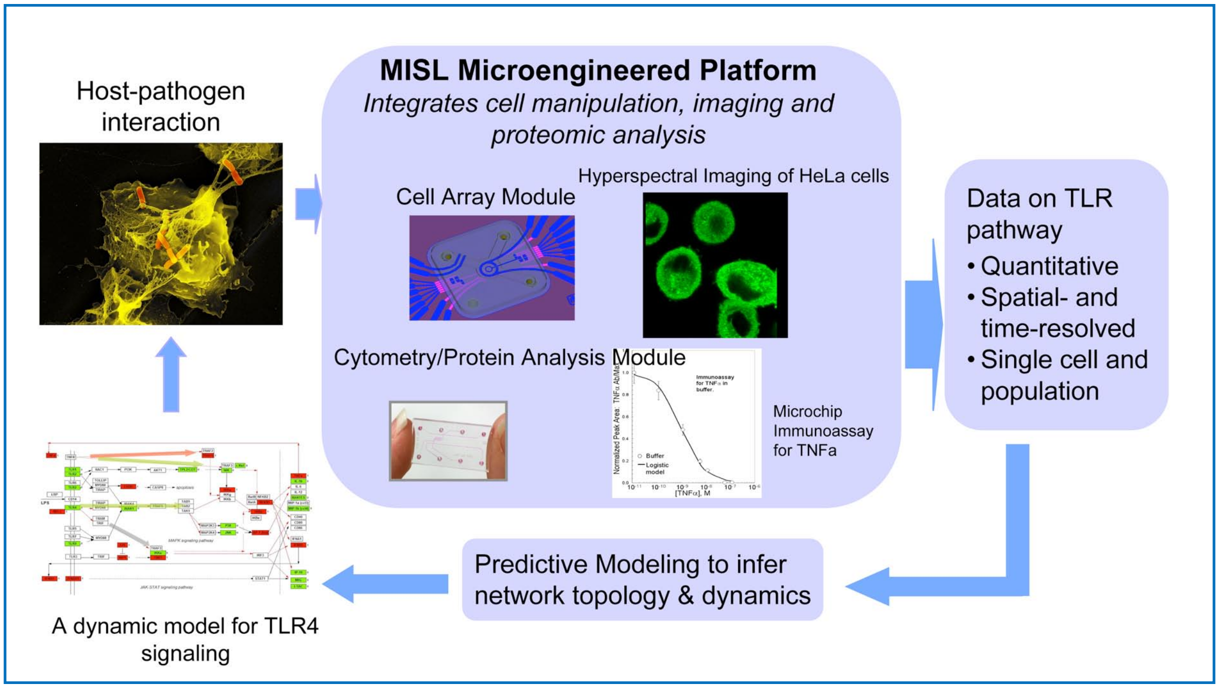
The work completed in **Project 95214, Terahertz Microelectronic Transceiver (TμT) System**, for example, brought about a powerful, compact, and robust chip-level-integrated terahertz microelectronic transceiver (TμT) that will benefit national security missions in areas such as nonproliferation assessment, homeland defense, and compact tools to support the warfighter.

Applications include scanning through opaque materials for concealed weapons or materials; detecting explosives and weapons of mass destruction; establishing secure high-speed communication links; and enhancing scientific instrumentation for upper-atmospheric chemistry and astronomical space-based telescopes.

Another project focuses on creating the first microengineered platform for molecular interrogation of the human immune response to pathogens. The work of **Project 95215, Microscale Immune Study Laboratory (MISL)** *(see next page)* will have a profound impact in biodefense, infectious disease, and public health research and development.

The team developed novel technologies to elucidate molecular mechanisms of the innate immune response in host cells to pathogens such as bacteria and viruses, including the mechanisms used by pathogens to subvert, suppress, or obfuscate the immune response to cause their harmful effects. They are designing an instrument that enables multiplexed measurements of single cells, individually or as part of a population, under well-controlled conditions – something nearly impossible with current bench-top technologies.

MISL's unprecedented sensitivity and multiplexing could revolutionize the way microbiology and immunology research is performed.



Project 95215, Microscale Immune Study Laboratory (MISL)

Creating an integrated single-cell manipulation and interrogation platform and predictive models provide molecular- and cellular-level understanding of innate immunity signaling pathways with unprecedented speed, resolution, sensitivity, and multiplexing.

Grand Challenges

Integrated Fiber Lasers for Efficient High-Power Generation

81752

D. A. Kliner, S. Moore, E. J. Skogen, B. Do, G. A. Vawter, A. V. Smith, W. J. Thomes Jr., W. R. Bolton Jr., R. L. Schmitt, G. R. Hadley, R. L. Farrow, J. P. Koplow, P. E. Schrader

Project Purpose

Despite great advances in spectral coverage and output power, high-power lasers remain predominantly laboratory tools that are bulky, fragile, inefficient, and/or provide poor beam quality. Massive investments (billions of dollars) in various laser technologies have failed to overcome these limitations, and numerous potential applications of lasers have thus been rendered impractical or impossible. These applications span all Sandia strategic management units and are central to national defense, homeland security, nuclear weapons, energy surety, and economic competitiveness.

We are developing a versatile, new laser technology capable of simultaneously meeting the stringent combination of optical and physical requirements for practical high-power lasers. The approach is based on our breakthroughs in power scaling of rare-earth-doped fiber lasers, a technology in which Sandia holds a leadership position. Although radical power scaling is a significant objective, the Grand Challenge (GC) encompasses the more ambitious goal of integrating nearly all of the laser-system components into a rugged, monolithic fiber-optic platform. This research leverages and greatly extends other Sandia optical expertise and capabilities (in microlasers, pump diodes, waveguides, nonlinear optics, numerical modeling, and system engineering) to enable miniature, ultraefficient, high-power devices that will revolutionize the application of lasers to real-world problems.

The GC research project comprises five primary technical areas:

- rare-earth-doped fibers and fiber amplifiers (including pumping methods and output-beam formatting)
- pump diodes
- microlaser seed sources
- waveguide and laser/amplifier modeling
- system engineering and testing.

FY 2006 Accomplishments

We demonstrated 84 percent coupling efficiency from a diode bar to a 10-fiber array (exceeding the milestone of 80 percent). Diode bars are the most cost-effective source of high pump power, and Sandia's side-pumping method is the only approach capable of coupling the raw, unformatted diode-bar output into a fiber. For comparison, the conventional approach using formatted diode bars provides a net coupling efficiency of about 50 percent, resulting in a factor of 3 increase in wasted pump power (16 percent vs. 50 percent); the conventional approach also increases the cost (by a factor of about 5), complexity, and size of the pump source.

We demonstrated > 1 MW peak power with diffraction-limited beam quality. We demonstrated > 1 MW in FY 2005, but the beam quality was not diffraction-limited. High beam quality is required for many applications, and we met the milestone of maintaining the high peak power while improving the beam quality to the theoretical limit. The corresponding in-fiber peak irradiance was 440 GW/cm^2, a world record.

We demonstrated > 1 mJ pulse energy with diffraction-limited beam quality. Applications such as remote physical sensing (three-dimensional imaging ladar) are often more sensitive to pulse energy than peak power. A target of 1 mJ was chosen based on DoD and intelligence ladar application needs. We demonstrated 1.1 mJ, again with diffraction-limited beam quality (another key ladar requirement). The corresponding in-fiber fluence was 410 J/cm^2, another world record.

We generated wavelengths between 213 and 4400 nm by frequency conversion of a pulsed fiber amplifier. Broad wavelength coverage is needed for chemical and biological detection, remote physical sensing, and secure free-space communications. We demonstrated efficient frequency conversion using a simple optical setup, generating watt-level powers from the mid-IR through the deep-UV.

We developed time-dependent numerical model of fiber amplifiers. Waveguide and laser/amplifier modeling underpins many of this project's activities. We developed a model that treats pulse propagation in a fiber amplifier with arbitrary refractive index and rare-earth-dopant profiles, including the effects of fiber bending (mode distortion and loss) and saturable gain. This model accurately reproduces the > 1 MW and > 1 mJ experimental results with no adjustable parameters.

We showed accurate treatment of self-focusing in a fiber amplifier. Self-focusing sets the ultimate upper limit on the peak power attainable from a fiber amplifier. Much confusion exists in the literature concerning the value of this limit and the behavior of a beam as it is amplified toward the limit. We developed a numerical model that treats self-focusing in fiber amplifiers with arbitrary refractive-index profiles and in bent (coiled) fibers. We showed that the beam propagates through a series of "stationary modes" as it is amplified toward the self-focusing limit. Furthermore, we showed that the self-focusing limit is nearly identical in a fiber and in a similar bulk material, even when the fiber is coiled.

Significance

Our technical accomplishments resulted in world record achievements in fiber laser performance. Our External Advisory Board stated, "The contributions of modeling, coupled to experimental results, have been especially impressive." We exchanged visits with major defense contractors and made a significant effort to publicize the results of the project. As a result, our work is getting recognized in the science and technology community.

This project is providing opportunities for growth in a new research capability at Sandia. As a result of our accomplishments, we are developing a space-based laser communications concept, several groups at Lockheed Martin submitted a proposal to cooperate with us on further fiber laser development, and Sandia was invited by a Defense Advanced Research Projects Agency program manager to submit an idea for a new generation of laser designators.

Refereed Communications

R.L. Farrow, D.A.V. Kliner, P.E. Schrader, A.A. Hoops, S.W. Moore, G.R. Hadley, and R.L. Schmitt, "Bend-Loss Filtered, Large-Mode-Area Fiber Amplifiers: Experiments and Modeling," in *Proceedings of the Solid State and Diode Laser Technology Review*, June 2006.

R.L. Farrow, D.A.V. Kliner, P.E. Schrader, A.A. Hoops, S.W. Moore, G.R. Hadley, and R.L. Schmitt, "High-Peak-Power (> 1.2 MW) Pulsed Fiber Amplifier," in *Proceedings of Photonics West*, p. 61020L, January 2006.

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Advanced Fusion Concepts: Neutrons for Testing and Energy

81753

D. B. Sinars, J. S. Arzigian, A. B. Baker, T. E. Drennen, T. L. Sanders, P. J. Griffin, G. A. Rochau, C. J. Garasi, D. J. Dorsey, R. L. Coats, R. W. Lemke, E. J. Parma Jr., S. A. Slutz, W. J. Tedeschi, J. R. Schendel, C. M. Craft, K. J. O'Brien, D. M. Fordham, J. D. Rogers, D. L. Hanson, M. P. Desjarlais, T. A. Haill, D. R. Welch, B. V. Oliver, A. C. Robinson, R. A. Vesey, R. B. Campbell, P. J. Christenson, M. Jones, R. R. Johnston, J. W. Kellogg, L. E. Ruggles, C. L. Ruiz, D. F. Wenger, R. J. Lipinski, M. F. Young, A. J. Suo-Anttila, J. T. Ford, B. B. Cipiti, J. E. Kelly, M. A. Sweeney, J. T. Cook, J. H. Saloio Jr.

Project Purpose

The purpose of the project is to discover and understand pulsed-power-driven advanced z-pinch fusion concepts that can eventually be used to generate neutrons for testing, transmutation, and energy. We are studying the likelihood of various advanced z-pinch concepts to produce sufficient neutrons for the desired applications, using both computational models and experiments. For each potential application, we are developing the neutron yield and flux requirements that must be satisfied for the application to be feasible.

FY 2006 Accomplishments

We made substantial progress in all areas of the project during FY 2006. A number of relevant experiments were conducted on the Z facility that are providing validation data for the computational models under development. These experiments utilized diagnostics and cryogenic infrastructure developed as part of this project. We developed several computational frameworks and models that are being tested against experimental data acquired as part of the project. These models will ultimately be used to predict the pulsed-power requirements needed to meet the neutron source requirements of the various applications.

We also made great strides in refining the potential applications for advanced fusion sources. Calculations with the MCNP and MCNPX neutronics codes show that an externally driven neutron-multiplying assembly using a z-pinch neutron source (Z-EDNA) is feasible and may meet some Defense Programs (DP) testing needs. We created a preliminary design for a z-pinch-driven transmuter with the purpose of burning transuranic waste from light water reactors. We developed a concept for a chamber to contain the blast and gases from a 10 MJ yield demonstration in ZR. We developed a model of economic feasibility for US energy production with a transmutation option assuming a successful z-pinch transmuter.

Significance

Success in discovering and understanding new advanced z-pinch concepts for fusion neutrons would have significant impact on the High-Energy-Density Physics & Inertial Confinement Fusion (HEDP/ICF) program if the sources proved to have lower pulsed-power requirements to meet our application goals than our baseline approaches.

Success in developing a pulsed-power Z-EDNA source could provide a useful platform for DP testing that could ultimately replace other sources in the laboratory complex, most significantly, those using highly enriched uranium.

Success in defining a pulsed-power-based nuclear waste transmutation source could provide a roadmap for incorporating such a device into Sandia's nuclear energy programs and enhance Sandia's stature in the national nuclear energy arena.

Sources that reduce the pulsed-power requirements for given fusion yields would ultimately be attractive as inertial fusion energy sources and could help make pulsed power a more attractive path to fusion energy.

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D.B. Sinars, D.F. Wenger, K.L. Keller, J.E. Bailey, G.A. Rochau, and J.L. Porter, "Focusing Adjustable Spectrometer with Temporal Resolution (FASTR) for the Sandia Z Facility," to be published in *Review of Scientific Instruments*.

D.F. Wenger, D.B. Sinars, G.A. Rochau, J.E. Bailey, J.L. Porter, A.Y. Faenov, T.A. Pikuz, and S.A. Pikuz, "Focusing, In-Chamber Spectrometer Triplet (FIST) for High-Resolution Measurements on the Sandia Z Facility," to be published in *Review of Scientific Instruments*.

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Highly Pixelated Hypertemporal Sensors for Global Awareness

95211

R. R. Kay, K. Wojciechowski, R. Lovejoy, D. V. Campbell, M. L. Holmes, J. E. Levy, T. M. Gurrieri, R. Chanchani, G. A. Keeler, P. J. Robertson, K. A. Sandquist, J. A. Mercier, D. K. Serkland, S. L. Shinde, D. D. Chu, J. L. Rienstra, T. P. Swiler

Project Purpose

The principal component at the focus of next generation remote sensing and surveillance systems is the focal plane array (FPA). FPAs drive many of the key limitations for current and future systems to provide true persistent global awareness. The most demanding applications require sensors with multiple tens of megapixels, operating at high sample rates. While state of the art is approaching the ability to provide FPAs of sufficient spatial dimension and pixel count, data rate for these very large arrays is currently limited to tens of samples per second per pixel by digital bandwidth at chip interfaces, and by packaging and thermal constraints. Even if these interfaces were improved, total data volume would overwhelm any traditional data handling system, given the stringent power, weight, and volume constraints in satellite applications.

This Grand Challenge project focuses on rethinking the limiting technologies and approaches related to fast-sampling operation. Among the challenges are developing systematically distributed data reduction techniques implemented at the pixel level, at the FPA level, and ultimately at nearby distributed processing nodes directly adjacent to, or as a monolithic part of, the FPA. Rethinking the whole architecture of modern sensor systems demands careful re-examination of the limiting technologies associated with highly integrated digital and analog electronics: dense packaging, radiation hardness, programmable digital and analog processing circuitry, power and volume efficiency, and thermal management; all areas where investment in Microsystems and Engineering Sciences Applications (MESA) would profit next-generation sensor systems. This project will define an FPA architecture, clearly define technology roadmaps, and demonstrate key enabling technologies, which will lay the foundation to realize a 2048 x 2048 pixel, 10,000 sample per second data rate FPA within three years of this project's completion. This is accomplished through research in four key areas:

System Design: This research area defines missions and utility of technology, resulting in specifications to guide design and development of an FPA.

Circuit Design: This research area will result in architectural, signal processing, and circuit solutions to implement FPA requirements in the demanding space and power constraints imposed by remote sensing systems.

Packaging Design: This research area will result in packaging technologies and assembly processes that will enable the assembly of large, gapless, FPAs with integrated signal processing. The approaches under investigation will allow for a modular approach in which large FPAs can be assembled from smaller, more producible pieces.

Optoelectronic Data Interconnect: An optoelectronic data interface composed of VCSELs (vertical-cavity surface-emitting lasers) and fiber optic cable is being developed. Integration of such a high-speed (10 gigabit per second) digital interface to an FPA will represent a major achievement. It will represent a fundamentally new way to move high-sample-rate data from an FPA to a downstream signal processor.

FY 2006 Accomplishments

We established candidate system architectures defined by three mission areas, and derived key focal plane requirements. A few central themes became clear:

- The minimum FPA size of interest is 2K x 2K pixels
- A spectral response band from 0.4 to 2.5 micrometers will cover the majority of applications
- Maximum sample rates can be reduced to 10 kHz and still satisfy most mission areas.

We documented the conceptual requirements, along with a supporting spreadsheet for performing system-level trades to evaluate impacts on FPA specifications.

Additionally, we completed a detailed computer modeling environment which allows detailed study of clutter noise resulting from optical line-of-sight jitter and spacecraft orbital motion while imaging a structured scene. This tool can be used to study the signal processing necessary to reduce this noise level to below the background random noise level.

We reviewed relevant technologies in the literature, many recommended by the External Advisory Board. This background information has proven valuable in evaluating how the mission space investigated in this project relates to other ongoing work, and how approaches considered by others may apply to our research.

Architectural Design
We focused on pixel level architecture and completed a comprehensive survey of analog-to-digital conversion architectures to aid the team in trades involving this important piece of the signal processing.

Four candidate architectures have emerged; all involve implementation of filtering at the pixel level. Two involve analog pixel processing and two involve analog-to-digital (A/D) conversion and digital domain processing. Physically, the architectures require multiple silicon layers to implement a traditional scanning architecture, with multiple A/D converters located on silicon layers beneath the pixel field and an additional event-driven scanner capable of scanning multiple windows of interest.

We completed two pixel unit cell and event-driven pixel readout circuit designs, including electrical simulation and physical layout.

Packaging
We began with a thorough survey of silicon stacking and hybridization technologies. Based on this foundational information and system concepts from the system and silicon circuits teams, baseline packaging requirements and a modular assembly approach have emerged.

High Speed Interconnects
We completed a detailed analysis of interconnect options for focal plane arrays. A decision to integrate VCSEL optoelectronic interconnects on the FPA has been made based upon the complexity of designing and fabricating multi-gigabit per second electrical interfaces.

Significance

An External Advisory Board (EAB) with members of academia, government, industry, and the user community was assembled, and two review meetings have been completed. The board verified the importance, uniqueness, and approach of this research.

The Remote Sensing and Verification Program (RSVP) has been developing innovative solutions to national security space monitoring programs since the mid-1960s. The RSVP program led the nation in developing systems of these types, but as with any technology, we are beginning to see fundamental limitations of current approaches. In most cases, the limiting sensitivity of our systems is related to three interrelated principal factors: sensitivity, spatial resolution, and temporal resolution. All other factors being equivalent, sensitivity improves with finer ground sampling and signature analysis is improved with finer temporal sampling. Therefore, to improve the sensitivity and characterization our systems can achieve, we are pushed toward systems that operate faster and simultaneously have finer ground sampling. In most cases, technology supporting these mission domains cannot be found in the existing industrial base. It is expected that success of this research will lead to new remote sensing capabilities for the defense and possibly civilian space communities.

Other Communications

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Terahertz Microelectronic Transceiver (TμT) System

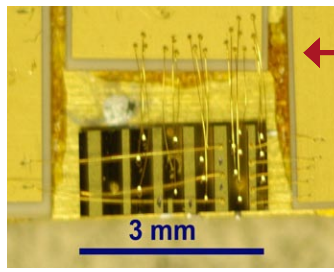
95214

M. C. Wanke, R. E. Jorgenson, I. Waldmueller, W. W. Chow, T. A. Barrick, C. Schmidt, J. Nogan, A. M. Sanchez, R. E. Allman, P. A. Miller, A. Gin, F. Leonard, C. D. Nordquist, M. Watts, J. J. Hudgens, E. W. Young, W. Pan, W. J. Zubrzycki, A. A. Talin, R. J. Foltynowicz, I. Brener, J. L. Reno, M. Lee, C. Highstrete, C. T. Fuller, S. Samora, A. D. Grine, E. A. Shaner

Project Purpose

The underused terahertz spectral region offers an unprecedented, disruptive advantage to enable new national security missions in areas such as nonproliferation assessment, homeland defense, and compact tools to support the warfighter. Applications for highly integrated terahertz microelectronics transceiver (TμT) systems include imaging through opaque materials to scan for concealed weapons or materials, high-specificity chemical detection of explosives and weapons of mass destruction proliferation, lightweight airborne remote detection platforms, and secure high-speed communication links.

Replace This:
- Molecular gas FIR laser
- Size: ~ 1 m x 0.3 m, 25-75 kg
- Fragile precision alignment
- One wavelength at a time
- 40 mW at 2.5 THz



With This:
- Semiconductor THz QC Laser
- Size ~ 2 mm x 0.1 mm, 1 g
- No mechanical parts
- Scalable to mass production
- Multi-wavelengths straightforward
- 50 mW at 3 THz, 250 mW at 4 THz

Six QCLs integrated on one chip

The technical goal of this Grand Challenge project is the development of a chip-level-integrated TμT. This powerful, compact, and robust device will be a key enabling subsystem immediately adaptable to several national security mission areas. The TμT Grand Challenge capitalizes on our existing lead in terahertz quantum cascade lasers (QCL) and harnesses Sandia's strengths in several other critical areas (including terahertz signature measurement, innovative terahertz detectors, microwave system expertise, microelectronic integration and packaging) and brings them to bear on this single enabling subsystem.

FY 2006 Accomplishments

The main thrust of the project is the development of the terahertz microelectronic transceiver (TμT), which will ultimately integrate new terahertz QCLs with terahertz mixer detectors. Towards this end, in FY 2006 we fabricated and validated a first set of QCLs operating at 2.8 THz. We also designed and built a Schottky diode mixer receiver that will be tuned for use with these QCLs, making the first QCL/Schottky terahertz receiver.

Designs of the experimental components needed for the first TμT demonstration were completed and are being constructed. A parallel effort is identification of the terahertz spectral signatures of materials of interest to national security. In FY 2006 we improved both spectral measurement quality and theoretical understanding of the terahertz absorption by energetic material vapor in the frequency range covered by the initial TμT components.

In response to External Advisory Board recommendations, we also designed a new broadband spectrometer for future measurements of relevant terahertz signatures. The new spectrometer will deliver sufficiently high spectral resolution to take proper advantage of the very narrow linewidth offered by a QCL driven TμT. This will result in spectral signature identification with very high material specificity and discrimination.

Also, we investigated incipient but potentially disruptive materials and device physics and made several advancements in the understanding of the terahertz electrodynamics of nanostructures, including nanotubes, nanowires, and quantum wells. In the case of quantum well terahertz detectors, this improved scientific understanding translated directly to a dramatic improvement in the sensitivity of a terahertz quantum well prototype detector.

Finally, we began modeling and simulation work towards design of an integrated TμT.

Significance

To date terahertz technology has been mostly limited to laboratory demonstrations with a few large and expensive systems in actual field use. The latter is mostly dominated by astronomical instruments. This limitation has been greatly influenced by the size, complexity, and cost of existing technology – primarily the difficulty in making high-power compact sources.
The recent development of a solid-state source has changed the whole outlook for terahertz technology and is enabling not only advances in compact sources, but whole new compact system concepts that may radically open up new applications.

Areas where immediate application may occur is compact flight systems for upper-atmospheric remote detection of molecules with scientific and national security interest, as well as combining with existing ion mobility spectrometers for enhanced material identification. Longer-term prospects include short-range imaging through various materials such as envelopes or clothing for inspection capabilities.

More compact systems may also enhance scientific instrumentation for upper-atmospheric chemistry and astronomical space-based telescopes.

Successful development of a TμT system is the core advance that will generate disruptive new capabilities in a wide range of application areas. Sandia already has most of the individual pieces needed to have a major impact here. This Grand Challenge project provides the mission cohesion, system focus, and "pump-priming" needed for Sandia to become the world-leader in terahertz technology and system development.

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I. Waldmueller, W.W. Chow, E.W. Young, and M.C. Wanke, "Linear and Nonlinear Responses of Intersubband Lasers," presented at CLEO 2006, Long Beach, CA, May 2006.

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E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, "Tunable THz Detector Based on a Grating Gated Field-Effect Transistor," in *Proceedings of the SPIE Photonics West*, p. 612006-1, January 2006.

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W. Pan, "Experimental Studies in a Two-Dimensional Quantum Dot Array Sample," presented (invited) at National High Magnetic Field Laboratory, Tallahassee, FL, April 2006.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, J.L. Reno, and S.J. Allen, “Terahertz Detection Using High-Mobility GaAs-AlGaAs Heterostructures,” presented at Materials Research Society Spring Meeting, San Francisco, CA, April 2006.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, and J.L. Reno, “Solid-State Microelectronics for the Terahertz Spectrum,” presented at American Institute of Engineering Conference on THz Systems III, San Diego, CA, November 2005.

M. Lee, M.C. Wanke, E.A. Shaner, A.D. Grine, and J.L. Reno, “Terahertz Microelectronics: New Components and Applications,” presented at Southeastern University Research Association Symposium on THz Applications, Washington, DC, June 2006.

Microscale Immune Study Laboratory (MISL)

95215

A. K. Singh, C. A. Apblett, A. Martino, S. Branda, C. Branda, C. D. James, M. B. Sinclair, K. L. Sale, J. F. Poschet, K. D. Patel, B. Carson, S. L. Rempe, N. Srivastava, T. Perroud, M. W. Moorman, R. P. Manginell, S. J. Plimpton, S. B. Martin, J. Joo, A. E. Herr, R. Rebeil, T. W. Lane, D. M. Haaland, J. M. Faulon, S. M. Brozik

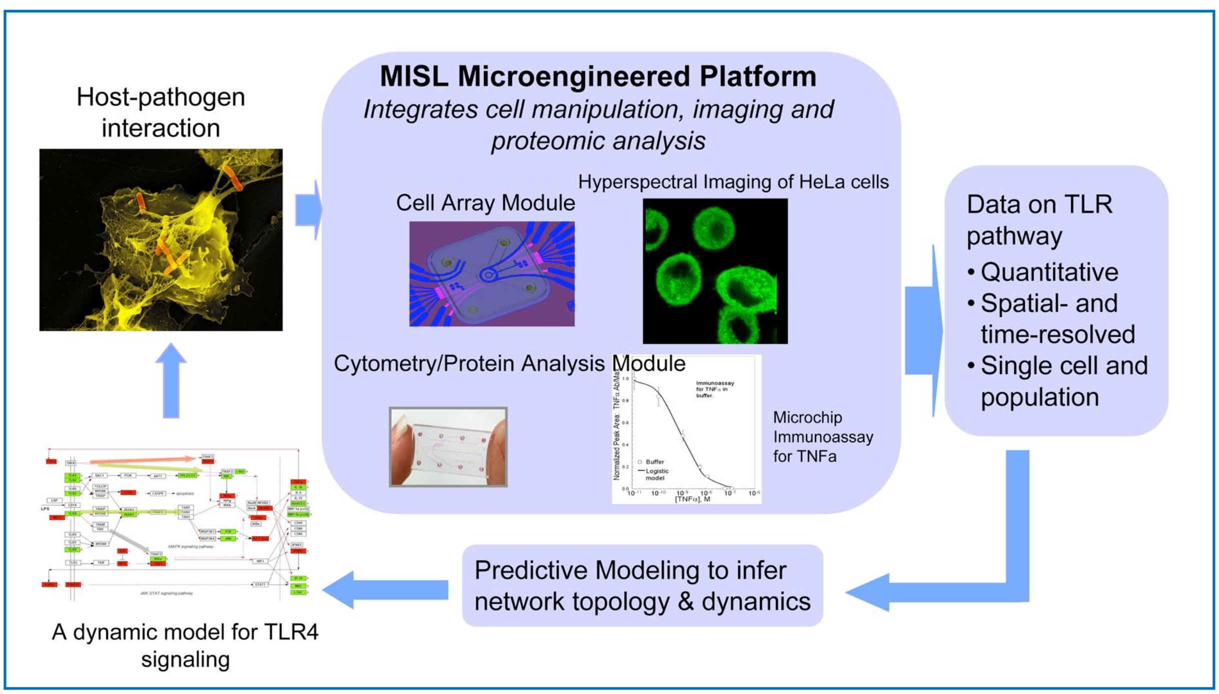
Project Purpose

Our overarching goal is to develop novel technologies to elucidate molecular mechanisms of the innate immune response in host cells to pathogens such as bacteria and viruses, including the mechanisms used by pathogens to subvert/suppress/obfuscate the immune response to cause their harmful effects.

While considerable progress has been made towards understanding signaling pathways of the innate immune system, such as toll-like receptor (TLR) signaling, serious gaps exist in our knowledge. For example, the interconnections between pathways, the interactions between proteins, and the modifications to proteins that occur during signaling are incompletely understood. Moreover, the existing signaling pathway models lack both quantitative and kinetic information. Furthermore, existing techniques do not provide detailed spatial information (e.g., localization) regarding the signaling events inside a cell.

To develop a molecular- and cellular-level understanding of the innate immune system, protein concentrations and reaction kinetics of pathway activation must be quantified at the single-cell level. Cells in a population do not all have the same initial physiological state or degree of infection. Consequently, measurement of the cellular signaling pathway is not accurately represented from data averaged over a population of cells. Further compounding the challenge are feedback characteristics (primary vs. secondary signaling) inherent to the innate immune system, which can be lost in population-averaged measurements. Thus, to construct a cellular-level understanding of the innate immune response, an ensemble of responses – measured at the level of the individual cells – is needed.

Our approach aims to tease apart the response of the TLR4 signaling pathway by quantifying this



Creating an integrated single-cell manipulation and interrogation platform and predictive models provide molecular- and cellular-level understanding of innate immunity signaling pathways with unprecedented speed, resolution, sensitivity, and multiplexing.

signaling network in macrophage and epithelial cells challenged with lipopolysaccharide (LPS) and pathogenic bacteria. Through development of a high-throughput measurement and analysis instrument capable of measuring protein concentrations, states, and interactions in host cells, we will obtain temporal, spatial, and cell-state information comprising the basis of the innate immune system. Specifically, the instrument design enables multiplexed measurements of single cells, individually or as part of a population, under well-controlled conditions – something nearly impossible with current benchtop technologies. Validation of the system is achieved by integrating data generated through benchtop biological experimentation and information acquired through use of the nascent microengineered platform with predictive models.

In short, we propose to develop an integrated high-throughput experimental and computational approach that provides "system-level" quantitative spatiotemporal data with single-cell resolution for the TLR pathway.

FY 2006 Accomplishments

Experiments this year focused on: 1) establishing proof-of-principle results, 2) preparing and acquiring biological reagents, 3) developing the components of the microfluidic platform integral to high-throughput experimentation, 4) validating hyperspectral imaging for cells of interest (macrophages and HeLa) and 5) building computational models and simulation tools. Major accomplishments for the year are:

- Developed biological hypotheses and experimental plan to test them.
- Benchtop experiments completed for measuring TLR response to LPS chemotypes.
- Fluorescent reporter constructs developed/acquired in macrophages and HeLa cells.
- Overall platform architecture defined.
- Designed, fabricated, and tested first-generation components of the integrated platform:
    - on-chip cell cytometry demonstrated and compared with a commercial cytometer
    - on-chip cell sorting and immunoassays demonstrated
    - on-chip electrophysiological measurements demonstrated.
- Hyperspectral imaging applied to interrogate macrophages and HeLa.
- Developed a refined signaling + regulatory TLR pathway model.
- Developed models to predict potential protein-protein interactions.

Biology Core Accomplishments: We made significant progress in developing fluorescence reporter constructs in macrophages, *Yersinia pestis* and *Francisella tularensis*. We are also using our wet-bench monitoring techniques to carry out a comprehensive analysis of the TLR4 network response to different doses of *Escherichia coli* LPS. We are also beginning to work with LPS that we have purified from our pathogens of interest; we will use our wet-bench techniques to gain early indication as to whether different LPS chemotypes stimulate the TLR4 network in different ways. Specifically, we have performed western assays and cytokine profiling to monitor the key nodes of the TLR4 network.

Platform Core Accomplishments: We designed, microfabricated, and tested chips for carrying out flow cytometry, cell sorting, and proteomic analysis. We implemented flow cytometry of tracer particles and macrophage cells in a prototype microfluidic device. Flow focusing was accomplished using a hydrodynamic mechanism and cell throughput was demonstrated as low as 1 cell/s and up to 210 cells/s. Development of an optical trap-based cell selection and sorting system enabled selection, manipulation, and low-throughput sorting of macrophage cells. The actuation method is based upon the same principles used in conventional laser tweezer studies. We also made progress in developing electrophoretic immunoassays for analysis of secreted and intracellular protein content. Chip-based immunoassays were developed for TNFa, IL-6, and iNOS. Current work is being performed on exogenous protein (spiked) in buffer and cell culture supernatant to test detection antibodies and generate rough sensitivity estimates. Sensitivity is currently at low nanomolar to high picomolar concentrations.

We also used hyperspectral imaging to 1) image oscillations of the transcription factor NFkB in HeLa cells carrying RelA-GFP constructs and 2) image

RAW264.7 cells to determine autofluorescence. Significant advances in our imaging visualization and multivariate curve resolution (MCR) hyperspectral analysis software were implemented to improve the speed of data collection and analysis.

Computational Core Accomplishments: A predictive model was developed to predict potential protein-protein interactions based on literature data. We also extended the existing TLR network models to include signaling events at the protein level as well as gene regulatory pathways.

Significance

Innate immunity is the human body's first line of response against a bacterial or viral invasion. Subversion of innate immunity is a strategy common to numerous pathogens, especially emerging pathogens. A systems-level understanding of the TLR pathway will enable improvements in early diagnosis of infection. Further, we anticipate that an improved understanding of the TLR pathway will reveal molecular targets for development of effective prophylaxis and therapeutics. Project success will have a profound impact in biodefense, infectious disease, and public health research and development.

In the area of microsystems research and development, Sandia has internationally recognized facilities and expertise – perhaps the best in the world at present. Over the past 10 years, we have also established formidable expertise in biological and computational sciences. MISL combines these three areas to develop tools to revolutionize basic research in host-pathogen interaction. In other words, MISL would create the first microengineered platforms for molecular interrogation of the human immune response to pathogens. Current technologies inherently lack spatial and temporal resolution; in most cases requiring large amounts of cells for analysis (thereby losing quantitation and stochasticity) and because of a lack of multiplexing, current systems do not provide a system-level view of the molecular events. Hence, there is a fundamental need for high-sensitivity, highly multiplexed laboratory analytical tools that will improve our understanding of interactions between pathogens and hosts at the molecular level.

By exploiting Sandia's microsystem expertise, MISL aims to achieve unprecedented sensitivity (tens of cells) and multiplexing (by integrating chips for cell imaging and flow cytometry), thereby making current benchtop procedures obsolete. A research endeavor of this scale puts Sandia in a strong position to develop such capabilities and, potentially, revolutionize the way that microbiology and immunology research is conducted.

Refereed Communications

A.V. Hatch, A.E. Herr, D.J. Throckmorton, J.S. Brennan, and A.K. Singh, "Integrated Preconcentration-SDS-PAGE of Proteins in Microchips Using Photopatterned Cross-Linked Polyacrylamide Gels," *Analytical Chemistry*, vol. 78, pp. 4976-4984, July 2006.

T. Perroud and K. Patel, "Rapid Fluorescence-Activated Cell Sorting with Optical-Force Deflection in a Microfluidic Device," in *Proceedings of the Micro Total Analytical Systems 2006*, November 2006.

MEMS Core Design for iSMART

100327

B. R. Rohrer, R. H. Olsson, D. K. Kholwadwala, K. A. Peterson, S. L. Hingorani, J. W. Wheeler, D. R. Sandison, P. C. Galambos, S. A. Whalen, C. A. Apblett, S. Buerger

Project Purpose

A primary purpose of this LDRD project was to develop a microelectronic system with cybernetic applications. We evaluated several application areas within biomedicine. We chose an implantable neural interface based both on its technical challenges and potential impact. After taking stock of the current technologies that would be used in such an interface, we focused our technical development efforts on developing a novel electrode. The other microelectronic components that would be integrated into an implantable microelectronic neural interface are more technologically mature than the electrode itself. Our goal was to develop an electrode capable of stimulating the human peripheral nervous system (PNS) and recording neural signals from the PNS as well.

Neural interfaces with the PNS have so far proven to be limited in their performance. In order to be effective in humans, neural interfaces must have a long functional lifetime, be minimally invasive, and provide a certain amount of both feedback and feedforward signals to the nervous system. Existing electrodes all fail in at least one of these categories. The electrode we planned to develop would allow all the conditions for feasibility in humans to be met.

FY 2006 Accomplishments

Electrode selection

We reviewed a variety of interfaces to peripheral nerves described in scientific literature, and identified cuff electrodes as having the potential to provide long-life and high-performance. Other electrode types were deemed inherently damaging to the nerve, and thus undesirable for use in healthy humans.

We identified the extent to which previous research had developed cuff electrode technology. In particular, the simulation of nerve excitation has been very limited, due to the complexity of the computational processes involved. In addition, we noted that individual advances in cuff technology had not all been integrated into a single solution. These findings indicate that there is a clear path for Sandia to make a unique contribution to electrode technology. By using Sandia's computational resources to simulate novel electrode placements and geometries, and by incorporating such newly discovered high-performance electrode arrays with existing advances in cuff technology into a single package, we could leapfrog previous cuff electrode designs.

Simulation

We employed simulation capabilities to better understand how electrode design and stimulation waveform affect the recruitment ability of cuff electrodes. While extensive investigation of a variety of designs was not possible, we did successfully conduct three-dimensional simulations of a cylindrically symmetric nerve trunk with a tripole electrode configuration, reproducing the qualitative results reported in the literature.

The simulation was conducted with COMSOL, a commercial finite element analysis that specializes in solving systems that contain coupled physics. In the case of the nerve trunk, current passing through the electrodes stimulates the nerves to fire. When the nerve fires, it creates a small electrical potential that may also be detected at the electrodes. This type of coupled physics is difficult or impossible for typical solvers to address. Using COMSOL, we are creating the first nerve simulation capable of performing both stimulation and recording.

Fabrication methods

We began obtaining samples of silicone and experimenting with methods of joining and metallizing. We are poised to begin fabricating and testing electrodes.

Significance

The preliminary design work we conducted in FY 2006 is significant in that it provides a roadmap to advance the state of the art of neural interface technology, specifically neural interfaces to the peripheral nervous system. PNS interfaces are notable in that they allow measurement from and stimulation of well-understood neurons. In contrast to interfaces with central nervous system, the effects of neurons in the PNS are clear. Development of an interface to the PNS is, in essence, the low hanging fruit of neural interface technology.

During the course of this project, we developed professional relationships with the MIND Institute, the BRaIN Center, and the Psychology Department at the University of New Mexico. These relationships will give us access to neuroscientists, neurophysiologists, neurosurgeons, and psychologists, as well as world-class neuroimaging and electrophysiology lab equipment for follow up projects.

A high-performance interface with the PNS that is suitable for use in humans opens up the door for dramatic improvements in prosthesis technology. Such an interface would allow an amputee to "feel" objects that came in contact with their prosthetic limb, as well as to control that limb with unprecedented dexterity. If a PNS interface became sufficiently noninvasive that it would be acceptable for use in healthy humans, then an entire class of human augmentation applications becomes feasible. People with physical weakness could be assisted by mechanical devices, and workers performing heavy manual labor could employ cooperative cybernetic assist devices, for example. Perhaps one day, military personnel could control unmanned vehicles or aircraft by thought while maintaining a natural sensory representation of the vehicle's status.

Machine Perspicacity Feasibility Study

100736
F. Rothganger, D. E. Small, G. S. Davidson, K. W. Larson

Project Purpose

Human-like machine discernment is an extremely difficult, unsolved problem that prevents us from solving many national security problems. For example, previous Grand Challenge LDRD projects addressing augmented cognition, border security, and virtual perimeter systems were limited by our current capabilities in machine discernment. The purpose of this project was to evaluate the technical feasibility and programmatic needs of a future autonomous machine perspicacity system that combines Sandia's high-performance computing (HPC), remote sensing, and information analysis expertise. The vision is to develop the world's most advanced machine discernment system, capable of not only recognizing, locating, and tracking thousands of static and dynamically moving objects in their native environments, but also understanding the object's intent, recommending a course of action, and even taking "limited" action by itself to reduce margins of error.

Even with significant advances in computing power over the past 40 years, current machine perception systems are still greatly constrained by available desktop computing capabilities. This project will assess whether substantial improvements in machine perception can be made by using Sandia's world-class high-performance computing clusters to simultaneously process gigabytes per second of raw sensor data from multiple sensor sources (e.g., vision, radar, ladar, hyperspectral, etc.). Based on past experience in parallel computation, we believe a massively parallel implementation may allow exploration of radically new adaptive machine discernment algorithms with substantially improved performance. Within this project, we will identify technology gaps and one or two high impact technology focus problems, and evaluate the feasibility of creating an autonomous perspicacity machine that will impact multiple national security applications, such as remote sensing and DOE physical security.

FY 2006 Accomplishments

We interviewed potential users of this technology to learn their needs, which include:

- Storage, retrieval, and object recognition in image sets.
    - an accelerated supercomputer imaging database for the imagery analyst
    - application guided hardware compression for image intelligence
    - change detection in air and ground video
    - feedback driven perception
- Detection of events and suspicious activities in real-time.
    - scalable video for large scale situational understanding
    - computer vision for suicide bomber detection
    - cognitive assistant for video analysis

We developed concepts for four of these:

- An Accelerated Supercomputer Imaging Database for the Imagery Analyst: This concept combines a large-scale database implemented on a supercomputer with imagery feature-extraction, object recognition and machine learning. It enables new imagery analyst (IA) tools to rapidly search through terabytes of real-time and historical imagery, returning a ranked list analogous to the "Google" method of returning the most likely candidates first.
- Application Guided Hardware Compression for Image Intelligence: This concept dynamically compresses images, such that tasked areas receive little or no compression, and nontasked areas receive a variable amount of compression based on their potential intelligence value. The system combines two different analyst-defined sources of information to determine the level of compression. One is a multiresolution map of the world that specifies the expected intelligence value of each pixel. The other is a set of feature detectors that pick out items of interest to the analyst. The software tool used to view imagery can profile the analyst's actions and infer which

areas are interesting. It can then update the ranking of that portion of the earth's surface, and also train the classifiers to hunt for regions with similar appearance. The analyst can also directly set the values of certain regions (tasking) and explicitly select objects to train on.

- Computer Vision for Suicide Bomber Detection: This concept extracts a real-time three-dimensional (3D) skeletal model of a subject walking using passive, multibaseline, camera 3D imaging, and then analyzes the gait dynamics. It compares this against an ideal model for the subject's estimated height and weight. Significant deviation from the ideal model could be an indication that the subject is carrying a large mass around his or her waist or back. It also compares the gait against the dynamics of specific types of personnel-worn suicide vests/belts.
- Cognitive Assistant for Video Analysis: This concept builds an artificial cognitive assistant to support military personnel whose job is real-time video analysis. We envision that the warfighter who uses this system is stationed at a terminal where his or her primary task is scanning real-time video from unmanned aerial vehicles for anthropomorphic activity that could be considered suspicious. Users train the system to look for certain types of objects (vehicles and pedestrians) and spatiotemporal patterns in the live video stream. The system then marks any such patterns on the screen that the user appears to be missing.

Significance

We developed concepts that emphasize key elements of a larger design for a machine perspicacity system. Such a system would run on a high-performance computer, process multiple inputs, scan for significant objects/events, and remember what it has seen. This system would communicate with each user through an interface that focuses attention on the most significant information, and which learns from the user what is important. It is the hope of the project team that in the near future Sandia will consider a single, focused project to develop such a system. The need for this capability has been established by our research, and Sandia is well-positioned to pursue this as a leader in the computational field.

Corporate Investments

Corporate Investments ensures a progressive future and guarantees Sandia's place as a world leader in science and technology. It creates the opportunity for disruptive and revolutionary science and technology that anticipate future mission space and future research and development needs.

Corporate Investments focus on Advanced Concepts, promote excellence in science and technology, and aid in training and acquiring the next generation of outstanding scientists and engineers.

Advanced Concepts projects explore potential solutions to future threats that might dominate the national security agenda in the next 10 to 20 years.

The Seniors' Council (a group of Sandia senior scientists and engineers) seeks to support truly new and innovative ideas that do not yet have a place in defined areas of investment. The scientific merit of the new concept must be very high. The new concepts typically originate from an existing area of expertise, but lead beyond, or cut across, existing investment areas. The goal of the council is to support the originator/advocate of the new concept.

For example, Project 103004, Modeling and Simulation of Spectra Expected from Radiation Sensors Made from Arrays of MEMS Scale Capillaries, is exploring a high-risk concept for detecting ionizing radiation using microscale tubes to hold the detecting gas at very high pressures, near liquid density.

The team is predicting the performance of such tubes and validating the computational models with experimental prototypes. If successful, pager-sized gamma-ray or neutron spectrometers could eventually be made with broad impact to NNSA, DOE, DHS, and others.

Investments also enable University Collaborations to create new science and engineering technologies, to employ complementary resources and skills for achieving common goals, and to recruit and guide top students for Sandia's work force.

Advanced Concepts

Mentor/PAL

93422
M. R. Glickman

Project Purpose

The purpose of this project is to research the use of physiological signals for understanding the performance of individuals and groups, particularly those who are under stress.

FY 2006 Accomplishments

We completed all three planned data-collection experiments, each involving 12 subjects. These experiments represent the first time electroencephalogram (EEG) data have been collected from subjects in a resting state, i.e., in which subjects are not required to participate in an active manner (this has been done for functional magnetic resonance imaging).

We successfully completed the first stage of analysis on the data collected to this point – application of the second-order blind identification (SOBI) blind-source separation algorithm. We began the second stage of analysis with the initial application of Granger causality analysis to a portion of the data, and presented a poster based upon the initial Granger analysis at the Human Brain Mapping 2006 conference.

We analyzed components of neural activity with respect to their stability over time. We identified Bayesian networks as a key technique for analyzing both EEG and the earlier physiological data. Bayesian networks not only provide for classification and analysis, but can also serve as generative models that allow us to explore the potential effects of adjusting independent variables (such as the rate of presentation of new material in a training system) to bring individuals to a higher-performing state.

Experimentation with the application of Bayesian networks to the earlier physiological data is proceeding well. And we prepared a draft manuscript documenting the EEG results.

Significance

This project is engaged in fundamental neuropsychological research that may broaden our understanding of cognitive functioning. In particular, we are focusing on the performance of individuals and groups under stress, which is important to national security. It also may provide a basis for further valuable activities, such as guiding training strategies to improve such performance or building technology that helps to optimize the performance of individuals or groups under stress.

Other Communications

M.T. Sutherland, Y. Zhang, M. Ding, and A.C. Tang, "Top-Down Versus Bottom-Up Processing in the Human Brain: Distinct Directional Influences Revealed by Integrating SOBI and Granger Causality," presented at Human Brain Mapping, Venice, Italy, June 2006.

Identification of Threats Using Linguistics-Based Knowledge Extraction

93423

P. Chew, S. J. Verzi, B. Mancke, T. L. Bauer, N. M. Rahal, G. S. Davidson

Project Purpose

The purpose of our project is to build an infrastructure that allows the contents of the world-wide web to be visualized at a high level, with particular emphasis on ideology. The aim is to classify or cluster documents by ideology based on their linguistic content or the words they contain. As at least a third of web pages are not in English, and ideologies of interest may be expressed in other languages, a subsidiary purpose is to develop a method whereby documents can be compared regardless of their language.

FY 2006 Accomplishments

We developed a new method for cross-language information retrieval (comparison of documents in different languages) and began to validate it. Our validation experiments shows that our method compares favorably with other published work. Based on our work, we filed a technical advance (which also documents the manner in which our approach to cross-language information retrieval allows for "noise reduction" through our choice of parallel corpora). Our method allows for documents in more than 2,000 languages (even little-used languages) to be compared.

Over a period of several months, we amassed a multilingual corpus of almost a million documents from the web, using web crawling techniques focused on finding "ideological" documents. This corpus is still growing as the web spider continues to run. The web crawler was implemented five months ahead of our originally anticipated schedule.

We began two types of analysis of the above-mentioned corpus: first, clustering of the documents to allow a high-level visualization of the universe of documents in terms of topics and ideologies; and secondly, predictive analysis to classify the documents into those that do and those that do not represent ideological threats. We developed the framework for these analyses and have an initial implementation.

Significance

We anticipate that our project will primarily benefit the intelligence community, specifically those who have an interest in addressing the open-source "information overload" problem. We aim to provide analysts a way of understanding the zeitgeist at the highest possible level and of seeing how individual web pages fit within the overall landscape of the internet (particularly with regard to the worldview, which might be encompassed in a particular page).

Our project allows a top-down approach to exploring the internet, compared to a bottom-up approach that relies on the analyst's knowing in advance what he or she is searching for. For much open-source material, existing tools such as Google might suffice for the latter approach.

An important technical by-product of our research that is likely to have applications in many areas is our technique for cross-language information retrieval, which relies on a computational approach using public-domain parallel multilingual text like a Rosetta stone. Our experimentation has shown that this approach provides extremely satisfactory results in predicting target documents that are already known to be translations of the source document; the percentage accuracy is very high, and we have significantly outperformed results reported in published research.

Refereed Communications

P.A. Chew, S.J. Verzi, T.L. Bauer, and J.T. McClain, "Evaluation of the Bible as a Resource for Cross-Language Information Retrieval," in *Proceedings of the Association for Computational Linguistics*, p. 7, 2006.

Other Communications

P.A. Chew and S.J. Verzi, "A Framework for Cross-Language Information Retrieval," presented at Computing Research Laboratory, New Mexico State University, Las Cruces, NM, 2005.

UV or IR Fluorescing Sensor for Explosive Material Components

93425
M. E. Welk, J. E. Parmeter

Project Purpose

We propose to develop materials that offer significant improvements in stand-off detection of explosive component vapors.

Materials that change their photoluminescent signatures in the ultraviolet, visible, or infrared range when an explosive volatile organic component is adsorbed to the surface will be synthesized to detect two classes of explosives: Comp B (a trinitrotoluene-based explosive) and TATP (triacetone triperoxide). When irradiated with the appropriate wavelength of light, these materials will fluoresce differently, providing stand-off detection of explosive compounds on surfaces such as cars or buildings. The adsorbing material could be deployed in paints or other coatings, or possibly on adhesive labels.

Detection of explosives is a critical ability in light of the increasing number of terrorist bomb attacks in the Middle East and elsewhere. Currently, reliable explosive detection is accomplished by swabbing a surface or sucking air past a filter and placing the swab or filter into an ion mobility spectrometer. This technique is extremely effective for close proximity detection (e.g., airport entry portals). However, an appreciable distance between the target and the filter renders this system useless. As only a few places are accessed through portal sites, there is a need to detect remotely the presence of explosives in open areas.

Stand-off explosive detection, wherein individuals and vital assets are physically separated from the severe damage resulting from an explosive detonation, is made more difficult by dynamic backgrounds, the necessity of a quick response time, and the need for high sensitivity and high specificity. This project was pursued in the hopes of obtaining proof-of-concept data on using photoluminescent materials for explosive vapor detection, specifically for TATP and other peroxide explosives.

FY 2006 Accomplishments

We reviewed TATP data in the first three months of the project and will soon present our spectroscopic data, thermal stability data, and physical characteristics in a SAND report.

We tested the photoluminescent signal of the solid state materials in the presence of TATP in a solvated state. In principle, the TATP ring should preferentially coordinate to the metal ions in the materials tested. In the case of crown ethers, such coordination has been shown to decrease the photoluminescent signal. In our solution-based tests, we detected no drop in photoluminescence for ZnS, TiO_2 or ZnSe. Infrared spectroscopy of solutions showed that in the acetonitrile solution containing 0.1 mg/mL TATP, the addition of TiO_2 eliminated the signals corresponding to the symmetric and asymmetric ring stretches of TATP.

Significance

The report on the characteristics of TATP will be useful to the explosive detection community. Although this explosive has been known for several years, reliable data on its spectroscopic characteristics, thermal stability, and physical characteristics have not been readily available. Further research ideas have been generated from the finding that the TATP ring cleaves in the presence of Ti^{4+}. If it is found that the fragments bind to the ion as predicted in the literature, this could be a useful scheme in other detection methods.

Other Communications

M.E. Welk, B.A. Simmons, and E.A. Cruz, "Synthesis of Photoluminescent and Photocatalytic Doped TiO_2," presented at Materials Research Society National Meeting, Boston, MA, November 2006.

Adaptable Software for Advanced Human/Computer Systems

79738
A. M. Bouchard, C. M. Johnson, G. C. Osbourn

Project Purpose

The purpose of this project is to create (i.e., design and implement) a software environment that the user can adapt. Consider the following problems:

- An analyst wants to customize certain analyses provided by her software system, but either the system does not support customization, or it's difficult to understand how to make the system do what she wants.
- Her colleage wants to analyze data from a new database, but his existing analysis tools won't talk to the new database.
- A security system analyst wants to simulate a new threat, but his software simulation tool contains no algorithm for this new style of terrorist attack.

Each of these problems (and a host of others) has a single root cause: the software only knows how to talk to, process, and present data in the single way it was programmed. Any modification to the software must be made by programmers. The cost and time scale for changes directly impacts user productivity.

In this project we are developing a user-adaptable computing environment in which users can modify the environment themselves on-the-fly, specifically, the data that is accessed, how the data is organized, and the algorithms that can be run on that data.

Essentially, the user acts as the programmer without having to know the underlying programming language, specifying a task using powerful English-like verbs. The code to execute this task is self-assembled "under the hood" using our self-assembling software technology. With security and intelligence analysts' needs in mind, we are designing and building the capability to analyze any kind of tabular data, intuitively and easily, and to build up more and more complex analyses for repeated use.

FY 2006 Accomplishments

The adaptable computing environment (ACE) can be divided into two large pieces from a design and implementation standpoint: user interface and under the hood. The user interface includes everything the user sees and interacts with. In the ACE, data and results are stored in books on bookcases rather than in files in a hierarchical tree of folders, as in the operating systems most widely used today.

We hired a graphic designer to illustrate the books, bookcases, pages, and so on, with near-photographic realism, and integrated these images into the user interface. This realism enriches the user interface objects, making them intuitive to use without training or documentation.

We designed and are implementing the interface using our self-assembling software (SAS) technology. The ramifications of this design are that it enables the user to create new books, bookcases, and other interface objects; easily modify, move, find, undo and restore data; and create English-like programs, because the interface itself self-assembles.

The design and implementation of the user interface required many months of work and tens of thousands of lines of code. We have filed two patent applications: one on the ACE interface, and one on the SAS design for that interface. The under-the-hood part of the ACE is everything below the user-interface layer.

One key accomplishment was the design and implementation of the auto-generation and self-assembly of executable code. The SAS generator not only generates and executes standard commands like math functions and searching, but can also self-assemble complex sequential or even hierarchical commands (subroutines). The SAS generator took 11 man-months to design and implement and resulted in more than 11,000 lines of code (in addition to 11,000 lines for the self-assembling building blocks themselves).

Another key under-the-hood accomplishment was the design and implementation of an auto-save and auto-load capability. This capability automatically saves new data, objects, and results to the hard disk, eliminating the need for the user to do so. It also loads all the data necessary to render and interact with interface objects on demand. Together, these capabilities enable the user to interact only with the entities on the interface (books, bookcase) and not worry about data names or locations on the hard disk.

In summary, we made significant progress in both the user interface and under-the-hood pieces, and integration of the two for a complete prototype is under way. We submitted 13 technical advances that cover many aspects of the English-like programming and innovative user-interface capabilities. We anticipate filing several patent applications on these inventions in FY 2007.

Significance

As we near the completion of a prototype ACE, we are preparing to test the software for usability. Our development has been aimed at security analysts and others who need to rapidly develop custom software in response to changing threats or problems that need to be solved. Our accomplishments this year bring us much closer to being able to address this unmet need of vulnerability and risk analysts, whether their problem domain is nuclear facilities, other infrastructure assets such as power facilities or the US Mint, intelligence information, or other problems of national security.

We are generating (and taking appropriate steps to protect) a great deal of intellectual property on this technology. Not only could the ACE enhance Sandia's leadership in vulnerability assessment and analysis for government customers, it could, if commercialize, also have significant technological and economic impact in the private sector.

Decision Support System Development Using Agent-Based Modeling

100337

G. A. Backus, J. D. Siirola

Project Purpose

Sandia has been developing and applying intelligent agent-based modeling technology for a number of years to enhance the capabilities of decision support systems (DSS). Recent Sandia work has focused on the agent-based modeling agent behaviors, optimality, and scalability. Recent Nobel Prizes in behavioral dynamics (McFadden, Granger, Stiglitz, Kahneman) and the first-ever Nobel Prize in agent-based modeling (Schelling) represent a new body of knowledge and methods that can be combined to lead to new, advanced, and robust capabilities for DSS relevant to many Sandia activities.

With this project we are testing the viability of combining these methods for DSS and working to ensure a scalable computation scheme. We are also demonstrating the utility of our new approach by developing an intelligent-agent model that will simulate health care decisions with multiscale interactions among local, state, and national populations.

Health care expenditures are consuming an ever-increasing portion of the gross domestic product. Decisions regarding health care are often made in a vacuum, as key decision makers have no decision-support tools to compare different recommendations. An effective agent-based tool for health care analysis would be an important asset in controlling spiraling costs. Combining economic and behavioral methods within an agent-based model will increase Sandia capabilities, creating a substrate for a wide range of decision support systems with broad applicability.

Specifically, we focused on demonstrating the simulation approach viability for each of the five critical components needed to develop the full system:

1. the scalable framework
2. the user interface to allow assessment with a multiscale system
3. a detailed aggregate disease-specific analysis
4. the agent-based version for the same model for validation and verification of the agent-based system
5. a policy simulation to verify flexibility of system to incorporate future requirements.

FY 2006 Accomplishments

We converted this model to an agent-based model using Sandia's ASPEN-HM system to test verification and validation methods and to develop an understanding of the modifications required when converting raw data for use in agent-based models. We disaggregated a subset of the model to determine the flexibility of alternative policy testing schemes. After designing a generalized multiscale frame work, we designed and completed a geographical user interface to allow fast evaluation of health-initiative impacts across multiple scales.

We worked with the University of Pennsylvania's Leonard Davis Institute and the various departments within the University of Texas (UT). Through their internal funding, they provided subject matter expert support and offered to develop text books and curriculum to support the effort should it move forward.

Dr. Kenneth I. Shine, the executive vice chancellor for Health Affairs at UT and the former president of the Institute of Medicine for the National Academy of Sciences, led a blue-ribbon-panel workshop to define the needs and specifications for the long-term development effort associated with this project. We also contracted with widely known modelers of health care issues, codeveloped a model of cardio-vascular disease, and tested the impacts of alternative care strategies under assumed financial constraints.

Significance

Current security interventions (e.g., Iraq and Afghanistan) show the need to add the social behavior component to decision making and strategic planning. Technological (military) solutions succeed or fail in the context of the social-behavioral environment. This effort expands Sandia's capability to include social-behavioral impacts within DSS.

The national medical community and various medical experts have recognized not only the need for this work but also our capabilities in this type of work and our unique ability to produce such a tool.

Seniors' Council

MEMS-Based Arrays of Micro Ion Traps for Quantum Simulation Scaling

85513
M. G. Blain, C. P. Tigges, B. Jokiel Jr.

Project Purpose

The purpose of this project is to build and package a microfabricated two-dimensional (2D) linear ion trap design for application in quantum simulation experiments at Los Alamos National Laboratory (LANL). Goals were to microfabricate a revised trap design and build a custom, high-vacuum/ high-temperature trap package that is compatible with the experimental photonics, electronics, and data acquisition systems necessary for quantum simulations.

Our objective is to complete fabrication and custom packaging of 2D linear ion traps and test the traps in LANL's quantum simulation experimental apparatus.

FY 2006 Accomplishments

We developed a custom ion trap chip packaging process using a standard 100 pin PGA (pin grid array) package modified for housing the ion trap chips. This custom packaging process included: 1) the fabrication of a counterbore and a through-hole in the package base; 2) the gold metallization of the package backside and the sides of the counterbore and through-hole to provide electrical continuity from the back to the gold package base; and 3) the simulation and selection of a die-attach adhesive that would be compatible with the vacuum and temperature requirements of the quantum simulation experimental apparatus.

This custom modification was successfully demonstrated. Additionally, a metallization process was designed for post-die-attach evaporation of gold onto the chip and package backs and fronts prior to wire bonding.

Significance

The development of this custom ion trap chip packaging technology is a significant advance in the application of microfabricated ion trap chips to quantum simulation and quantum information processing approaches based on trapped ions. This is an enabling technology that increases the number of applications of Sandia's ion trap chip technology.

Diffusionless Fluid Transport and Routing Using Novel Microfluidic Devices

89669

D. Reichmuth

Project Purpose

Microfluidic devices have been proposed for "lab-on-a-chip" applications for nearly a decade. Despite the unquestionable promise of these devices to allow rapid, sensitive, and portable biochemical analysis, few practical devices exist. It is often difficult to adapt current laboratory techniques to the microscale because bench-top methods use discrete liquid volumes (e.g., classically test tubes and beakers, or in the context of a modern automated laboratory, robotic pipetters and microwell plates, which handle microliter-size discrete volumes). Current microfluidic devices employ (typically nanoliter) streams of liquid confined in a branching network of micron-scale (effectively one-dimensional) channels. The liquid streams are never fully compartmentalized and there is no ability to make individual packets of material that can be transported or processed independently. In other words, there is always mixing of the materials in the channel along the length of the channel, and generally across the branches of the network.

We are demonstrating the feasibility of devices on-chip that will allow the division of a fluid stream into discrete packets moving controllably within the microchannels, and the independent control (creation and destruction, transport, sorting, mixing) of the packets on-chip. Each packet would then be equivalent to a minute test tube, holding a fraction from a separation or an aliquot to be reacted. Such a device would overcome the current barriers to implementing true multistep fluidic processing on a chip and allow us to escape layout and timing constraints on implementation of microseparation imposed by diffusion and channel cross-talk. The segmented flow could also be used for biological research, as cells could be isolated or grown within the fluid packets. We believe that the device concept we propose could have a major impact on microfluidic chemical analysis and enable new generations of flexible integrated microfluidic devices that would be much closer to the lab-on-a-chip ideal.

FY 2006 Accomplishments

We are currently working on the first two milestones: the demonstration of packet formation using a microvalve and the lossless transport of a packet in a microfluidic chip. Currently, we are examining the use of polymer-based microchips, as they could be cheaper and quicker to manufacture than our original glass devices.

Our tests with existing glass chips from a previous project show promise, as we showed packet creation using an in situ fabricated microvalve. However, these chips were designed for on-chip high-pressure liquid chromatography (HPLC) separations, and the dimensions of the channels are far from ideal for 2-phase packet generation. In FY 2006, we investigated the use of elastomeric polymers for the channels, as well as plumbing and valving of the flow. We created a new capability at Sandia for rapid prototyping and fabrication of polydimethylsiloxane (PDMS)-based microfluidic devices.

We are also taking advantage of the ability to created segmented flow using Sandia-designed microfluidic connectors. These connectors allow the testing of passive segmentation schemes. For example, we have shown that an analyte peak can be segmented by a fluorinated hydrocarbon. The segments can be analyzed for an arbitrary amount of time. We showed that we can extend the analysis time by a factor of five, which has the potential to greatly increase signal to noise. Also, diffusion between packets cannot occur, so long-term storage of nanoliter-sized volumes is possible.

Finally, we used the high diffusion rate of oxygen though PDMS to attempt to grow bacteria inside a PDMS chip.

Significance

Our key technical accomplishments were the development of two liquid-phase microfluidic chips and the establishment of rapid elastomeric chip prototyping at Sandia. These accomplishments can be leveraged to create novel microfluidic devices for infectious disease research or homeland security applications.

Other Communications

D.S. Reichmuth, “Microvalves for On-Chip Pressure Injections and Fluidic Routing,” presented at Symposium on BioMicroNano Technologies and Medicine, Albuquerque, NM, November 2005.

D.S. Reichmuth and E.B. Cummings, “Segmentation for Enhanced Detection, Storage, and Routing of Analytes,” in *Proceedings of the MicroTAS 2005*, p. 1, October 2005.

Emulsion Technology for Sample/Contaminant Collection

90498
C. J. Bourdon, M. E. Welk

Project Purpose

We studied a novel emulsion interfacial collection and transport technique that is applicable for a wide range of materials from small molecules and nanoparticles to cells and bacteria. Emulsion-based collection is particularly effective for proteins, viruses, and similarly sized analytes. This technique takes advantage of the affinity of these materials to hydrophobic/hydrophilic interfaces to efficiently trap, concentrate, and transport them. Because the affinity for adsorption to interfaces is ubiquitous, the emulsion preconcentrator can be applied to a broad spectrum of biological and chemical materials of interest in homeland security and water surety applications.

This study investigates the promising capability of emulsions to collect and concentrate materials from aqueous solution. Initial studies focused on demonstrating the feasibility of the emulsion interfacial extraction concept and then examining basic technological challenges for each stage of the process: emulsification of the mixture; diffusion and trapping on the interface; then combining the emulsion droplets to concentrate and collect. We then proceeded to focus on resolving issues involved in incorporating emulsification, collection, and coalescence into a single prototype system. The efficacy of this system to remove contaminants from solution was analyzed.

FY 2006 Accomplishments

This fiscal year, we successfully proved the ability of emulsion interfaces to effectively collect and trap materials from aqueous solution. We tested two aqueous systems. The first was a solution of 200 ppm bovine serum albumin (BSA), a common negatively charged protein. The second was coal bed methane produced water (CBMPW) from the San Juan Basin. This complex waste-brine mixture is a by-product of methane production in northwestern New Mexico, and Sandia is actively seeking methods to cheaply and efficiently clean it up so that it can be recycled.

Using a pendant-drop technique to dynamically monitor the interfacial tension, we tracked the adsorption of materials at the oil-water interface. Once the interfacial tension had stabilized, the aqueous phase was flushed with pure water while monitoring the interfacial tension. If the interfacial tension remained constant, this indicated that the material was irreversibly adsorbed and could be effectively removed from solution with the emulsion. Both the BSA and CBMPW showed large changes in the interfacial tension as molecules from aqueous solution adsorbed onto the oil-water interface. For the CBMPW, water chemistry analysis leaves us unclear exactly what chemical compounds are extracted from the water. Upon dilution of the aqueous phase with pure water, the BSA remained primarily adhered to the interface, resulting in no change in the interfacial tension. For the CBMPW, there was a slight increase in the surface tension, but the interfacial tension remained significantly lower than for pure oil and water.

This year we also built a prototype system to test the emulsion interfacial extractor concept. We created a small reservoir that could hold 200 mL of aqueous phase. An emulsion was created by introducing the oil phase through a small, stainless steel square-tipped needle (0.33 mm inner diameter). At low flow rates, the oil created large drops that did not provide much mixing with the aqueous phase. At higher flow rates (30 mg/s), the oil created a turbulent spray of droplets that caused vigorous mixing with the aqueous phase. For the BSA system, protein assay showed a progressive decrease in the residual BSA concentration as more oil was pushed through the aqueous phase.

With the CBMPW, it was difficult to directly determine what was being removed from the system. Total dissolved solids is one method used to measure the “dirtiness” of the produced water, but that will also account for the dissolved salts (mainly NaCl). We compared the performance of the interfacial extractor to filtration of the CBMPW using a 0.22 micron Durapore membrane filter. The interfacial extractor performed favorably compared to the membrane filter with both techniques showing ~ 10 percent decrease in the total dissolved solids. Our interfacial extractor technique has advantages in that unlike a solid filter, the oil phase is recovered while the contaminant remains trapped at the oil-water interface.

Significance

Our project sought to test a novel idea to use oil-water interfaces in an emulsion to trap and remove contaminants from one of the phases. We successfully demonstrated that emulsions do trap materials and we built a prototype device to apply the emulsion interfacial extractor concept. There are many fields where removing and/or collecting samples is important. We applied our technique to the clean up of coal bed methane produced water, which is one example in the energy applications area. This technique could also be used as part of sample collection for sensors or as a part of wastewater processing.

This technique has many similarities to traditional solid-filter technologies, such as carbon filter technologies. Both use heterogeneous interfaces to adsorb materials out of solution. However, the emulsion interfacial extractor has a very clear advantage – these systems are easily cleaned. When the interfaces are full of contaminant, the emulsion can be collapsed into the two immiscible phases, concentrating the contaminant and recovering clean liquids. This will generally happen spontaneously. Solid filters are generally disposed of or have to be regenerated at high temperatures or with chemical treatments (creating hazardous waste in some cases). Since our emulsions have dynamic interfaces, they are easily regenerated, leaving behind all of the contaminated interfaces.

Bioagent Detection Using Miniaturized NMR

90506
T. M. Alam, H. Fan, J. D. Williams, D. P. Adams

Project Purpose

The purpose of this project was to advance the development of a miniaturized nuclear magnetic resonance (NMR) detector by coupling advances in microelectomechanical systems (MEMS) fabrication with nanoparticle amplification. The ultimate goal was to produce a low-field NMR device that is portable and can be used in remote and noncontrolled environments. The advantage of microNMR is that detection is possible for a range of sensing environments, including optically opaque solutions such as blood, milk, or effluent. We evaluated two separate MEMS technologies for the fabrication of microNMR detection coils (between 1 and 750 nanoliters). The relative sensitivity of these detection coils, along with the technical limitations, was determined. We also pursued the fabrication of different sized nanoparticles, along with the conjugation nanoparticles to biological ligands (antibodies).

FY 2006 Accomplishments

We made considerable progress on the planned milestones.

The fabrication and testing of the NMR microcoils at low magnetic-field strengths has been completed. This testing showed that both MEMS designs have their limitations and advantages. For example, the solenoid design produces the optimal signal to noise at larger detection volumes, as well as providing the best configuration for implementation into a microdevice at the chip level. Unfortunately, the coils were time consuming to fabricate, and scaling of the device to smaller sizes resulted in skin-effect limitations due to the extremely thin films used in fabrication (5 micron thick films).

A generalized theory for the response of NMR coils in this thin-film regime was produced. On the other hand, the flat spiral coil was easily fabricated, but had poorer signal to noise figures. In addition, the required configuration of this spiral coil with respect to the magnetic field is not optimal for future NMR-on-a-chip design.

Based on these results, the future sensor platform for microNMR was selected. Any future development work will require a solenoid coil design but use a MEMS fabrication technique that can give standing coil dimension of the order of 50 microns.

We demonstrated that NMR amplification is dependent on the size of the nanoparticle. At higher magnetic-field strengths, the smaller the nanoparticle the higher the signal amplification effect. The optimal size was found to be ~ 10 nm.

We established a protocol for attachment of prostate cancer antibodies to commercially available superparamagnetic nanoparticles. We were unable to directly extend this attachment protocol to Sandia-prepared nanoparticles.

Significance

The results of this research are significant in that they impact the microdetection mission at Sandia. MicroNMR is not restricted to optically clear environments, and can be used in such media as milk, blood, or turbid effluents. This research provides an alternative microdetection platform for use in Sandia devices.

The use of paramagnetic nanoparticle amplification is also significant in that this provides an example where the carrier fluid (e.g., water) becomes the amplifier agent in the final detection scheme. The demonstration of antibody conjugated nanoparticles is important because it provides the required selectivity for individual bioagent detection. These advances point to future microNMR sensor development for biodefense and homeland security.

Refereed

L.O. Sillerud, A.F. McDowell, N.L. Adolphi, R.E. Serda, D.P. Adams, M.J. Vasile, and T.M. Alam, "^{1}H NMR Detection of Superparamagnetic Nanoparticles at 1 T Using a Microcoil and Novel Tuning Elements," *Journal of Magnetic Resonance*, vol. 181, pp. 181-190, 2006.

Other Communications

T.M. Alam, D.P. Adams, C. Benally, H. Fan, A. Frequez, V.C. Hodges, K. Peterson, R. Torres, M.J. Vasile, J.D. Willimas, A. McDowell, E. Fukushima, and L. Sillerud, "MicroNMR and Nanoparticle Amplification for Bioagent Detection: A Progress Report," presented at 17th Annual Symposium for Advanced Materials – Micro and Nano Devices, Albuquerque, NM, October 2006.

Understanding the Materials Physics for an Alternative for PZT 95/5

102608
E. L. Venturini

Project Purpose

The primary objective of this foundational project is to understand the materials physics of an alternative for PZT 95/5, a material of interest to Sandia. Issues to be addressed include understanding of the various phase transitions from both a thermodynamic and a lattice dynamic point of view; how these transitions vary with composition and pressure; determination of complete temperature-pressure phase diagrams for selected compositions; and assessing and controlling hysteretic and relaxational effects.

We investigated a new composition of the proposed materials revealing pressure-induced phase transitions and we determined a tentative temperature-pressure phase diagram. We will perform high-pressure neutron diffraction measurements to determine the structure of pressure-induced phases. The results of this work should yield important insights into the nature of the various transitions. Work was also started on developing a scaling relation between composition and pressure as far as the phase behavior is concerned.

FY 2006 Accomplishments

We completed detailed studies of the electrical properties of a sample of interest and determined a temperature-pressure phase diagram for this sample. This diagram revealed complicated phase behavior, including large hysteretic effects, that we by and large understand. We also identified the symmetry of most of the phases.

We have also started work on a new composition selected to help us determine more fully the phase space for this class of materials, and to assess similarities and differences among different compositions – differences that may be important for potential applications.

To obtain a more complete crystallographic identification of all the phases, particularly those induced by pressure, we will perform high-pressure neutron diffraction experiments at the Spallation Neutron Source at the Argonne National Laboratory. This facility has excellent capabilities for the pressure range we are interested in.

Significance

This project is foundational and is intended to make sure we understand the materials physics of a new material being proposed as a better alternative for a material now in use. By understanding the physics, including the phase behavior and electrical properties of the new material, we can avoid any potential pitfalls that could otherwise be encountered later. Thus, the work being performed in this project will be highly significant for the successful implementation of this material in the future.

Modeling and Simulation of Spectra Expected from Radiation Sensors Made from Arrays of MEMS Scale Capillaries

103004

M. S. Derzon, R. F. Renzi, P. C. Galambos, J. D. Williams, B. A. Simmons, S. B. Martin, R. P. Kensek

Project Purpose

Detecting special nuclear material is not presently viable with pager-sized gamma-ray or neutron spectrometers that have credible concepts of operations. Such a capability would have a broad impact on national security needs for both DOE and the Department of Homeland Security (DHS) by aiding in the detection and interdiction of smuggled nuclear material. These needs could be met using high-pressure MEMS (microelectromechanical systems) capillary arrays (miniature gas chambers of high enough pressure to be liquid or near-liquid density).

We believe we can make acceptably sensitive gamma and neutron spectrometers using small high-pressure gas capillaries. Very high pressures can provide liquid or near-liquid density material providing high interaction probabilities. The purpose of this project is to obtain the critical understanding necessary to justify additional investment in what could be a unique mix of new and old technology.

This project is focused on the following specific goals/tasks:

- Simulate device spectra using the ITS series of codes, COG, and other tools as needed to clarify the effect of capillaries on device function.
- Fabricate device primitives in silicon (or substitute). Pressure test to failure and compare with modeled performance.

The key questions to be answered are:

- For the gamma detection and the neutron detection problems, what are the limiting conditions created by the capillary walls? Is there promise to this technique and if so where?
- Do the high-pressure devices or pixelated gamma devices meet the performance promise over other radiation detection concepts?

FY 2006 Accomplishments

We have achieved several noteworthy accomplishments for this project. First, we created a computer model for the calculation of neutron spectra in high-pressure capillaries. Second, we developed tools that allow us to determine how gamma angle of incidence may be obtained from pixelated radiation sensors. Third, we pressurized individual capillaries to failure. Finally, we modeled stresses in silicon devices to infer failure modes and pressures.

The results of the spectral modeling (neutron) compares well with published experimental results. Extrapolated to the devices, we envision performance as spectrometers should be within a few percent in energy resolution. Angular resolution for high-energy neutrons is highly spectrally dependent. Crude comparison to existing devices implies that angular inference may be made for devices of the same area as those that are presently used for count rate measurements only.

The calculation of angular convergence (gamma) show that within uncertainties, the incidence angle for incoming radiation (meaning location) will converge before full spectra can be obtained. The implication is that weapons of mass destruction (WMD) may be found first via imaging before spectroscopy (a very welcome advantage to the user).

Experimental testing of capillaries with an inside diameter of 150 microns provided failure testing to 50,000 psi. This is well in excess of what would be required. Modeling of individual capillaries is consistent with this and illustrates that individual capillaries are feasible. Ultimately, we believe that mass fabrication of both individual capillaries and aggregate devices is reasonable.

Significance

We believe that when fully developed, this technology would dramatically improve the ability of the DOE, NNSA, DHS, and the Department of Defense to meet the nation's radiological and nuclear detection needs with a common architecture. The NNSA Office of Nonproliferation Research and Development (NA-22) and the DHS Domestic Nuclear Detection Office have expressed preliminary interest in these concepts and their potential but would like to see the concepts matured further. We believe by bringing together the breadth of Sandia's science, technology, and engineering capabilities to address this critical national security need illustrates Sandia's role as a leader in transformational research for the nation. Our concepts offer numerous benefits over current detection technology for our missions associated with defense, counter proliferation, and WMD detection.

This project proposes to exploit and extend Sandia's expertise in modeling, nanoscience, microsystems, and nuclear physics to create a radically new solution for radiation detection. If successful, this work will contribute significantly to our understanding of how to use nano- and microscale technologies to create radically new solutions for problems beyond those typically considered. It would potentially provide a fundamental new radiation sensor approach that could lead to a range of new system configurations. These can then be deployed on numerous platforms. This would be a significant contribution to Sandia's mission to strengthen US national security and would position Sandia as the solution center of choice for the next generation of radiation detectors.

Ultrafast Nanolaser Device for Detecting Cancer in a Single Live Cell

103005

P. L. Gourley, R. G. Copeland, A. E. McDonald

Project Purpose

Emerging bio/micro/nanotechnologies have the potential to provide accurate, real-time, high-throughput screening of live tumor cells without invasive chemical reagents when coupled with ultrafast laser methods. These optically based methods are critical to advancing early detection, diagnosis, and treatment of disease. The first-year goals of this project are to develop a laser-based imaging system integrated with an in vitro, live-cell, microculture to study mammalian cells under controlled conditions.

Milestones include:

- design and build an in vitro, live-cell culture microsystem to study mammalian cells under controlled conditions of pH, temperature, CO_2, oxygen, and humidity on engineered material surfaces;
- demonstrate feasibility of integrating the culture microsystem with laser imaging and nanolaser flow spectrocytometry to carry out single-cell analysis; and
- demonstrate viability of cell cultures in the microsystem by showing that cells retain healthy growth rates, exhibit normal morphology, and grow to confluence without blebbing or other adverse influences of the material surfaces.

A motivating factor for this project is to overcome several technical problems with imaging in conventional culture flasks made of injection molded plastics that are convenient for cell culture but present severe limitations for high-quality optical imaging. Imaging is limited to reflectance mode and the range of transmission of light through polystyrene is limited by the absorption onset near 340 nm. Stress birefringence is a problem when using polarized light; the thermal differential index of refraction is 10x larger than that of glass and the refractive index of polystyrene is not matched to the oil immersion lens or oil. Further, the refractive dispersion is 2x that of glass. Thus, a microculture comprising high-quality optical surfaces is imperative for better images.

We also sought to eliminate technical problems with imaging under microscope cover slips on slides. Microscopy of live cells in cover slip/microslide cavities is hindered by cell blebbing (formation of membrane vesicles to maintain homeostasis with environmental changes) and in extreme cases, cell apoptosis. In initial experiments, we showed that it is possible to microfabricate sealed cell microchamber plates to minimize these effects to slow the formation of blebs and apoptosis by a factor of about 10, to the point where cells retain good morphology and are able to attach to the slide surface. These data show that live cells can be imaged in simple microfabricated cavities for short periods of time (few hours) without more complicated thermoregulation of culture.

However, we concluded that optimum results require a more sophisticated system for thermoregulation for maintaining cell viability over extended (> 2 hours) periods of time. Consequently, a live-cell culture microsystem with closed and open perfusion was implemented on a Zeiss laser scanning confocal microscope platform. This microculture system was developed using a modified version of the LaCon cell culture system for microscopy. This system allows improved optical access and use of the best oil immersion microscope objectives to allow a full range of optical techniques and higher standards of microscopic analysis of living cells. In addition, heated microscope stages and climate boxes were employed to allow observation of cells under stabilized temperature.

FY 2006 Accomplishments

We implemented a live-cell culture microsystem with closed and open perfusion and climate control on a Zeiss laser scanning confocal microscope. This system uses improved optics and the best oil immersion objectives to allow a full range of microscopic analysis of living cells.

We developed aseptic (sterile) techniques to minimize contaminating organisms in the microculture by setting up an autoclave system and sterile assembly hood that were specialized for microcomponents. The system was successfully used resulting in high sterility in the microculture.

We developed three types of perfusion: full flush, continuous, and microinjection. The full flush completely exchanges the media. Continuous perfusion was implemented with a minipump to slowly pass fresh culture media with controlled flow rate. Microinjection of small (~ 1 microliter) volumes of molecular probe dyes or chemical reagents was implemented.

We successfully seeded and cultured normal and cancer mouse liver cells from American Type Culture Collection (ATCC), TIB-73, and TIB-76, respectively. TIB-73 is a normal liver cell and TIB-76 is the chemically transformed version of the mouse liver cell line. After seeding, the cells indeed did adhere to the perfusion cover slips and maintained viability for several days.

To assess overall health of the cells, we developed a rapid imaging technique, “shadow phase contrast,” for very high contrast, three-dimensional images. The contrast is wavelength dependent and offers the possibility of inferring chemical information from cells without using molecular probes. The cells exhibit good adherence properties and show signs of healthy growth by well-formed intracellular organelles, nucleus, mitochondrial reticulum, and nuclear chromatin.

Additional imaging modalities, native fluorescence and mitotracker molecular probes were employed to determine the health of the adherent cells. The adherent cells show more internal cell structure than cell suspensions placed under cover slips on microslides because the adherent cells are very thin (0.3 to 2 microns), more spread out, and two dimensional. This more planar geometry facilitates optical access to the internal cell structure. This is a significant advantage for imaging live cells in microculture.

We observed that microinjection labeling was fast acting, very efficient, and represents a huge advantage over conventional labeling of suspended cells (requiring incubation for 20 minutes, spinning, rinsing, and resuspending cells). The mitochondria occur as filamentous networks near the cell center and were observed to move with time. This is indicative of a healthy respiring cell adapting to varying energy demands during cell growth. Also, little background fluorescence was observed and no special flushing of unbound dye was required. This is another advantage that occurs because of the thin confining dimension of the culture.

The microculture system was relatively easy to use, performed exceptionally well for growing adherent cell cultures, and permitted new kinds of experiments to be performed. The advantages stem from several enabling features. We have only begun to exploit the capabilities of the perfusion chamber and its numerous advantages over the flask system for culturing cells.

Significance

The microculture system was relatively easy to use, performed exceptionally well for growing adherent cell cultures, and permitted new kinds of experiments to be performed. We demonstrated a microculture cell system that could be integrated with other biocompatible microcomponents or materials for enhancing the bio/material interface or improved sensitivity or selectivity and could be used for new kinds of experiments for basic research in cell biology or biophotonics or for novel applications as biosensors.

Mitochondria are dynamic intracellular organelles that play a central role in oxidative metabolism and apoptosis. The recent resurgence of interest in the study of mitochondria has been fueled in large part by the recognition that genetic and/or metabolic alterations in this organelle are causative or contributing factors in a variety of human diseases, including cancer. Several distinct differences between the mitochondria of normal cells and cancer cells have already been observed at the genetic, molecular, biochemical, and biophysical levels. Certain of these alterations in mitochondrial structure and function might prove clinically useful either as markers for the early detection of cancer or as unique molecular sites against which novel and selective chemotherapeutic agents might be targeted.

Given the importance of mitochondria in the development of diseases such as cancer and respiratory failure, it is crucial to develop methods for detecting changes in mitochondria as a marker for early detection. Photonic techniques are expected to play a significant role because they have the capacity for ultrahigh-speed detection of large numbers of organelles and cells to permit accurate assessment of the statistical variations in a large population.

In addition, because the mitochondrial matrix is exquisitely sensitive as an environmental sensor that signals by changes in shape and optical signature, knowledge of the physical and chemical principles used in this highly adaptive biology can be envisioned as the basis for construction of a novel set of environmental and bioweapon sensors.

Refereed Publications

P.L. Gourley, J.K. Hendricks, A.E. McDonald, R.G. Copeland, K.E. Barrett, C.R. Gourley, and R.K. Naviaux, "Mitochondrial Correlation Microscopy in Single Cell Cancer Diagnosis," presented at Mitochondrial DNA and Cancer Epidemiology Workshop, Bethesda, MD, September 2006.

Hollow Waveguides for Instrumentation in Intense Radiation Environments

104949
J. D. Weiss, G. R. Iben

Project Purpose

The purpose of this work is to examine the susceptibility of hollow-core and other optical waveguides to ionizing radiation. All of the waveguides under consideration are suited to transmission in the mid-infrared region of the electromagnetic spectrum. There is reason to believe that they are relatively insensitive to such radiation, but testing must be done to verify that expectation. Testing is to be done at Sandia's Annular Core Research Reactor for exposure to neutrons and gammas, and at Sandia's Gamma Irradiation Facility for exposure only to gammas.

FY 2006 Accomplishments

We purchased hollow-core waveguides, silica waveguides, chalcogenide waveguides, silver halide waveguides, and "ZBLAN" waveguides (the acronym represents the combination of several elements comprising this material: zirconium, barium, lanthanum, aluminum, and sodium), infrared detector systems, lenses, and infrared light-emitting diodes.

We designed an optical system for the coupling of infrared radiation to the waveguides from the light-emitting diodes and to the detectors from the waveguides. Initial fabrication has begun.

Significance

If the expectation of radiation insensitivity is borne out, these waveguides could function in a high-radiation environment, such as that of a nuclear power plant or satellite, for communications or sensing.

Nanoporous Films for Epitaxial Growth of Single Crystal Semiconductor Materials

104953
H. Fan, D. B. Burckel, D. D. Koleske

Project Purpose

The purpose of this project is to explore aspects of crystal growth through confined growth masks. The well-understood kinetics of bulk crystal growth are modified when the crystal growth occurs in a confined space. When confinement occurs on a size scale commensurate with crystal growth kinetics, interesting macroscopic behavior results. For instance, heteroepitaxy, or crystal growth of one material system on a substrate of another material system, is a long-sought goal that will continue to increase in importance. Current research has shown that growth on patterned substrates can significantly reduce defect density when the pattern size scale is in the 10-20 nm range. Additionally, growth of magnetic material such as NiFe in a growth mask with pores on the size scale smaller than the typical grain size of ~ 50-80 nm reduces the probability of randomly aligning magnetic domains, leading to an increase in the coercivity.

These are but two of the many examples where the physics of crystal growth in a confined environment yields interesting, new, and useful behavior. It is worth noting that both of the size scales mentioned above are below that easily obtainable using standard semiconductor industry patterning techniques, and thus push the study of these phenomena to the margins, where e-beam writing and other serial processes are the only top-down options.

Our approach is to use nonconventional patterning techniques to generate growth masks with feature sizes of interest, pursuing bottom-up self-assembly such as surfactant templated silica and carbohydrate/block copolymer self assembly, as well as interferometric lithography. Furthermore, our goal is to pursue a variety of crystal growth methods, including vapor-phase epitaxial semiconductor crystal growth as well as electrochemical deposition and atomic layer deposition.

FY 2006 Accomplishments

We made progress in investigating electrochemical deposition of gold into high-aspect-ratio photoresist structures. Our initial experiments entailed using interferometric lithography to generate a two-dimensional rectangular array of holes with dimensions ~ 5 µm tall, 0.5 µm in diameter, and separation of ~ 1 µm on glass slides coated with a Ti/Au/Ti seed layer. Two different negative resists were chosen, SU-8 and NR-1. The parts were plated using a galvanic electrochemical bath, frequently used and optimized for plating parts in photonic band gap applications with dimensions > 1 µm. Thus the structures in this project represent a departure from the parts typically electroplated.

Because no prior baseline for the electrochemical deposition parameters existed, two sets of plating current density were selected, 6 mA/cm^2 and 1 mA/cm^2. Both of these parameters fall at the low end of the electroplating equipment limits. Nevertheless, we were successful at plating into both types of resist. After resist removal, significant areas of the patterned SU-8 sample contained uniform arrays of gold posts that were ~ 3.2 µm tall by ~ 0.5-0.6 µm in diameter. The NR-1 resist experienced some level of delamination upon immersion in the plating bath but selective areas of posts were obtained with height of ~ 2 µm and diameters ~ 0.4-0.5 µm.

This first set of experiments demonstrated several issues that we must address, such as adhesion of the resist to the substrate, resist uniformity, and particle control. Additionally, it is apparent that low current densities are required to achieve good morphology in the plated gold posts. These initial results are very encouraging and we expect that as we address these issues and improve the pattern uniformity and quality this research will proceed in several promising directions.

Significance

Although our results are preliminary, the ability to pattern and electrochemically fill high-aspect-ratio structures on size scales from 10-20 nm up to over 1 µm will have tremendous impact on both the science and technology community at large as well as Sandia and its customers. Arrays of gold posts generated by this approach have great potential as photonic structures because their size and separation are both on the scale of optical wavelengths. Knowledge gained from plating gold posts can be readily transferred to plaiting magnetic materials such as NiFe, yielding magnetic structures. In addition, patterned substrates with arrays of submicrometer gold posts also have enormous potential as a platform for surface-science behavior studies.

A MEMS-Based Thermoacoustic Engine

104955

C. A. Apblett, S. A. Whalen

Project Purpose

This project is intended to understand the limitations of thermoacoustic generation of power at the small scale. The idea of making a microelectromechanical system (MEMS)-scale thermoacoustic engine requires understanding the limitations of heat transfer and air viscosity at small dimensions. To learn more about this, we propose developing a small, prototype heat regenerator that can show the thermal to mechanical conversion of power in a small engine.

FY 2006 Accomplishments

We placed an agreement with Los Alamos National Laboratory for thermal modeling of the thermoacoustic engines, and ordered a mask for the initial fabrication of a set of regenerators. We investigated several fabrication techniques, and the initial designs will be built in Foturan glass that is stacked up to be 1 mm thick, with 10 μm thick walls for the regenerator.

Significance

This engine, if successful, can harvest heat from very low gradients (< 15 °C) and convert them into mechanical energy, in the form of a vibrating membrane. Transducing this to electrical energy can happen at a reasonably high efficiency. This will then allow the harvesting of several environmentally ambient heat sources (such as day/night heating/cooling of rocks, and black/white radiative exchange cooling) to be converted into small amounts of electrical power at usable voltages.

Development of Sample Preparation Methods for ChIPMA-Based Imaging Mass Spectrometry of Tissue Samples

104973

R. Behrens Jr., J. S. Schoeniger, R. J. Bastasz, S. P. Maharrey

Project Purpose

The purpose of this project is to develop the methods needed to prepare biological tissue samples for examination with imaging mass spectrometry (IMS) implemented on Sandia's chemical imaging precision mass analyzer (ChIPMA) instrument. Results from these measurements will enable us to grow new capabilities in proteomic-based development of new diagnostic tests, patient-based treatment therapies, new methods to combat and treat ovarian cancer, and new ways to understand pathogenic signaling pathways. The ChIPMA instrument has been developed to provide high-magnification chemical images of complex materials, such as organics, polymers, and explosives. This provides the ability to understand complex chemical reaction processes at microscopic spatial scales.

Using a Fourier transform ion cyclotron resonance mass spectrometer provides high mass resolution and high mass accuracy measurements, making it the only instrument in the world that has both high spatial and high mass resolution. This project will develop the methods to prepare biological samples for examination with this instrument. This will open new opportunities to understand reaction processes in biological systems and create the opportunity to develop biomarkers for disease based on an understanding of the underlying biochemical pathways.

FY 2006 Accomplishments

By the end of FY 2006, we have accomplished most aspects of the first three tasks:

1. Two types of samples will be used for development of the imaging methods. First, a protein standard sample will be electrosprayed on silver, gold, and indium tin oxide (ITO) substrates. Then a pattern will be etched in the substrate using an ion gun. This pattern will then be used to develop and evaluate imaging methods. Second, *Caenorhabditis elegans* will be used to develop and evaluate imaging of a well-characterized biological system. The extensive knowledge of the *C. elegans* system will provide a good basis for evaluation of the imaging methods. We have procured equipment to handle *C. elegans*.
2. We have procured and set up a Leica cryo-ultramicrotome. This will allow preparation of samples ranging from 5 nm to 20 microns. The ability to prepare thin samples will enable the development of secondary ion mass spectrometry imaging methods beyond the original goals of this project.
3. We have designed, fabricated, and tested the electrospray-based coating system. The first protein standard samples have been prepared with the matrix coating system.

In addition to the original tasks, we have also procured new higher-voltage power supplies that will increase the mass range of the ChIPMA instrument. This will increase the mass range of proteins that we will be able to analyze using the time-of-flight mass spectrometer in the ChIPMA instrument.

Significance

We have implemented new methods to prepare biological samples for chemical imaging with the ChIPMA instrument. This enables us to carefully control the preparation of samples for IMS analysis. These tools will let us develop and evaluate IMS methods for investigating biological processes within tissues and cells. If successful, this will be the first instrument to couple high-resolution spatial imaging with high mass resolution mass spectrometry to allow the identification of complex biological molecules as a function of location within a cell or tissue sample.

Using a cryo-ultramicrotome to prepare very thin samples will enable the development of new IMS methods. This will provide new methods to probe the reaction processes in biological systems. It will also provide new capabilities to probe the properties and reaction processes in a wide range of different types of materials.

The following are several examples of how these new tools may be applied:

- Evaluation of reaction processes in genetically engineered organisms. For example, *C. elegans* has been used to probe the role of RNA in regulating gene expression (Nobel Prize in medicine, 2006).
- Investigation of signal transduction pathways in infectious disease.
- Search for reliable biomarkers in cancer and neurodegenerative diseases.
- The design and development of processes for renewable energy sources (e.g., recalcitrant cellulose) or hydrogen storage systems (e.g., oxidation processes in metal hydride storage systems).
- The design of new energetic materials for the nation's future munitions needs. Future munitions must meet new performance, insensitive munitions, and safety and health requirements. To design these composite materials will require understanding and designing materials to control reaction processes on microscopic spatial scales. The methods developed in this project will provide unique methods to probe these processes.
- Evaluation of material issues in the aging nuclear weapon (NW) stockpile. More detailed information on materials used in the NW stockpile will provide an improved basis for developing stockpile surveillance methods.

University Collaborations

System Dynamics Modeling to Assist Regional Water Planning: Modeling the Nonmarket Value of Water

69156

V. C. Tidwell

Project Purpose

Fresh water is central to the vitality of our society; however, current demands are increasingly being met through unsustainable practices. Establishing new sustainable practices is complicated by the differing values placed on water by the disparate sectors of our society (industrial, agricultural, environmental). Integrated modeling tools are needed to assist in regional water planning and to facilitate public involvement in the decision process. We take a novel approach to community-mediated water planning, employing a cooperative modeling process formulated within a system dynamics decision framework.

This work is being performed cooperatively with the University of Arizona and the National Science Foundation Science and Technology Center for the Sustainability of semiArid Hydrology and Riparian Areas (SAHRA). SAHRA is a multi-institutional, multidisciplinary research center working to improve understanding of the hydrologic cycle, provide improved tools for decision makers, and raise the level of hydrology literacy. Through this collaboration, we gain access to extensive expertise and data from unique studies on valuation of water conducted in the Rio Grande and San Pedro River basins.

FY 2006 Accomplishments

A riparian valuation project for Aravaipa Canyon Wilderness in Southern Arizona continues in a second phase. Visitation data over a 14-year span have been prepared and linked to census socioeconomic data, environmental time series data, and a constructed travel cost variable. This project will increase the resolution of instream-flow recreational usage data, which likewise will be incorporated in the system dynamics framework for riparian value.

A bosque restoration survey instrument canvassed public values for the Middle Rio Grande corridor and recorded bosque recreational usage patterns. The survey was designed to supply new inputs for the riparian valuation module within the larger watershed decision support tool. Final survey responses have been coded and analysis is in process. Valuation of restoration allows improved analysis of water allocation in the Middle Rio Grande, results of which may be applicable to other southwestern areas with competing human and environmental usage.

Significance

Water scarcity has the potential to undermine the nation's economic, energy, and agricultural security. Significant tension exists over water allocations across international and interstate boundaries as well as the distribution of water among irrigators, urban developers, and environmentalists.

Our project provides data that allows direct comparison of water valuation between its use for irrigated agriculture, residential consumption, or in industrial applications. Specifically, the data and models developed from this project will be implemented within a decision support framework allowing decision makers, stakeholders, and the public to explore a wide range of water allocation strategies. Such models will provide the technical foundation for water resource decision support modeling objectives for Sandia's water security programs.

Other Communications

M. Weber and G. Woodard, "Shades of Grey: Modeling Tucson Area Residential Water Use," presented at the Arizona Hydrological Society, Flagstaff, AZ, September 2005.

M. Weber and S. Stewart, “Restoration Preferences for the Middle Rio Grande Bosque,” presented at the 5th Annual Meeting of SAHRA, Tucson, AZ, October 2005.

M. Weber and S. Stewart, “Restoration Preferences and Management for the Middle Rio Grande,” presented at the Universities Council on Water Resources, Santa Fe, NM, July 2006.

M. Weber, R. Berrens, and S. Stewart, “Quantifying the Value of Instream Flow in the Desert Southwest,” presented at the Natural Areas Conference, Flagstaff, AZ, September 2006.

Interactive Water Quality Modeling to Assist Regional Water Planning

69157
V. C. Tidwell

Project Purpose

Freshwater supplies are finite, and demand has tripled since 1950. This growing disparity between supply and demand necessitates difficult decisions that often directly impact groundwater levels and streamflows. Perturbations to water supply in turn may result in unexpected impacts on water quality and the riparian ecosystem. Unfortunately, making these decisions is complicated by the differing values placed on water by the different sectors of our society (industrial, agricultural, environmental, municipal).

While many models serve water resources managers in day-to-day operations, there is a lack of integrated modeling tools to assist in regional water planning and to facilitate public involvement in the decision process. We take a novel approach to community-mediated water planning employing a cooperative modeling process formulated within a system dynamics decision framework.

Water quality often limits the potential uses of scarce water resources in semiarid and arid regions. Consequently, changing water quality, particularly nutrient levels, is often an important concern with many water resource decisions. Chronically elevated nutrient loads may result in eutrophication of reservoirs and changes in riparian vegetation.

To best manage water quality, one must understand the sources and sinks of both solutes and water to the river system. Therefore, modeling efforts will need to include biogeochemical processes both within the river and within the shallow alluvial aquifers of the riparian corridor. Incorporating these processes and feedback is paramount to modeling and evaluating the water quality dynamics that may result from changing land use practices, waste water treatment policies, watershed/range management, and reservoir operations.

We are pursuing this work cooperatively with the University of Arizona and the National Science Foundation Science and Technology Center for the Sustainability of semiArid Hydrology and Riparian Areas (SAHRA). SAHRA is a multi-institutional, multidisciplinary research center working to improve understanding of the hydrologic cycle, provide improved tools for decision makers, and raise the level of hydrology literacy.

FY 2006 Accomplishments

We developed relationships between discharge, land use, and nitrogen sources and sinks using five years of synoptic sampling along a 300 km reach of the Rio Grande in central New Mexico. Average discharge in the river was significantly higher ($p = 0.01$) during 2001 and 2005 “wet years” (15.0 m^3/s, standard deviation [S.D.] = 0.88) than during the drought years of 2002 – 2004 “dry years” (8.85 m^3/s, S.D. = 1.77).

Wastewater treatment plants (WWTPs) were the largest and most consistent source of nitrogen to the river (1331 kg/day, S.D. = 19.8). Agricultural drain returns contributed less nitrogen than WWTPs in both wet (262 kg/day, S.D. = 54) and dry years (82 kg/day, S.D. = 38). Average total dissolved nitrogen (TDN) concentration downstream of the WWTP was 1.18 mg/L (S.D. = 0.22) in wet years and 0.52 mg/L (S.D. = 0.40) in dry years.

Possible explanations for the constant elevated TDN concentrations include continuous low flows, minimal channel vegetation, and large suspended sediment loads. Somewhat surprisingly, agricultural return flows had lower average nitrogen concentrations than river water originally diverted to agriculture in both wet (0.81 mg/L, S.D. = 0.17) and dry years (0.19 mg/L, S.D. = 0.18), indicating that the agricultural system is a sink for nitrogen. Lower average nitrogen concentrations in the river during the dry years are mostly due to the input of agricultural returns that comprise the majority of river flow in dry years. These findings imply that the hydrology of the river system is crucial in determining nitrogen removal rates in the river and agricultural system and must be included in the model. Furthermore, these changes in water source in the river during wet and dry years must be included in the model to accurately model nitrogen concentrations and cycling in the river.

Significance

Water scarcity has the potential to undermine the nation's economic, energy, and agricultural security. Significant tension exists over water allocations across international and interstate boundaries as well as the distribution of water among irrigators, urban developers and environmentalist. This project focuses on developing tools to better manage our limited resources and engage the public in the water planning process. In particular, this research will help quantify nutrient cycling processes and their impact on water quality.

Nutrients represent the leading anthropogenic source of pollution in surface and groundwater supplies. Nutrients are contributed to the environment through agricultural chemicals, urban storm and wastewater streams, atmospheric deposition and natural pathways. The chemistry and ultimate fate of these nutrients undergoes a complicated process of transformation, dispersion, and uptake in the environment.

Because of this complexity in the nutrient cycle, tools are needed to understand how changes in the natural and anthropogenic environment impact nutrient loads. In addition, tools are needed to assess how changes in nutrient loads impact the environment, how they may limit water use, and increase treatment costs.

Our work provides a comprehensive data set with which to explore the complexities of nutrient cycling in a large semiarid watershed. These data are being analyzed and modeled. The resulting relations are being implemented within a decision support framework allowing decision makers, stakeholders, and the public to explore a wide range of water use scenarios while understanding the downstream consequences in terms of water quality change. Such models will provide the technical foundation for water resource decision support modeling objectives for Sandia's water security programs.

Adaptive Algorithms for Use in the Rejection of Periodic Disturbances of Unknown Frequency

69166

E. S. Hertel Jr.

Project Purpose

The objective of this research with the University of Utah is to investigate algorithms for the rejection of unknown disturbances, with a particular interest in active noise and vibration control applications (ANC, AVC, or ANVC). Disturbances are assumed to be the sum of periodic signals with time-varying magnitudes, frequencies, and phases. The control algorithms are of the pure feedback type, where no reference sensor is assumed to be available to give a preview of the disturbance. These problems are more difficult to solve and less well understood. The algorithms are also able to handle systems with unknown dynamics that may change significantly over time.

Specifically, this work involves the development of new algorithms for adaptive disturbance rejection, with particular emphasis on the cancellation of periodic noise disturbances. In the development and implementation of these algorithms, we are using multichannel active noise control experiments to explore issues related to more globalized disturbance rejection, and we are examining the effects of adding additional sensors/actuators to multichannel configurations.

A key emphasis of this project is to treat unknown disturbances as well as unknown systems. Periodic disturbances with unknown frequencies may vary over a wide range. In adaptive feedback control, the rejection of such disturbances can be accomplished by using frequency estimation techniques or by deriving controller structures that obtain this estimate implicitly. Additionally, methods involving modified least-squares identifiers can be derived for identifying plant parameters online.

We are investigating these methods for the development and analysis of simple and efficient algorithms for systems where both disturbance frequency and system dynamics are unknown and possibly time-varying. The stability and parameter convergence of all algorithms considered will be examined through averaging theory.

FY 2006 Accomplishments

New algorithms for the rejection of periodic disturbances continue to be developed at the University of Utah. The focus on disturbances that are known to be periodic in nature makes this research relevant to many applications involving rotating equipment. Specifically, we are paying attention to narrowband adaptive feedback control, in which knowledge of the disturbance frequency and plant dynamics allows for perfect cancellation of periodic disturbances. We are considering algorithms that identify and adapt to variations in either disturbance or plant parameters. By assuming that the system dynamics have reached steady-state with respect to parameter update, simple and meaningful algorithms with established convergence properties have been derived.

Since many applications such as those concerning rotorcraft involve unknown and time-varying dynamics due to variations in environmental factors, we have developed methods for identifying plant parameters online. Several such methods have been implemented and investigated on an active noise control testbed at the University of Utah. A commonly used technique involves the use of conventional least-squares techniques to continuously update an estimate of plant dynamics.

By deriving a linear expression at the output of the plant, we found an estimate of the disturbance as well as the plant frequency response. This estimate is then used in determining the control input that exactly cancels the disturbance. Estimation of the parameters occurs continuously, whereas previous methods have required the collection of batches of data. We studied algorithms of both a continuous and discrete nature, and we implemented several different controller structures.

Based on this set of linear equations describing the plant output over time, we used recursive least-squares algorithms to achieve 97 percent attenuation of a sinusoidal disturbance acting on an unknown system through single-channel ANC experiments. We compared convergence rates between the various controller structures. Also, we investigated the ability to track both slowly and rapidly time-varying system parameters while maintaining considerable disturbance rejection. We used an exploration of various identification algorithms to reduce the effect of transients when rapid parameter variation occurs. The goal in this work is to obtain solutions that are viable in engineering practice.

Significance

There are many examples of applications where disturbance rejection is a primary control objective. Among these applications are a variety of engineering problems including the active control of noise in turboprop aircraft, the reduction of road noise in cars, headphones for noise cancellation, vibration reduction in helicopters, the reduction of optical jitter in laser communication systems, isolation in space structures of vibrations produced by control moment gyroscopes and cryogenic coolers, suppression of gearbox housing vibrations, and track-following despite eccentricity in disk drives and CD players.

In several of these applications the tracking of time-varying parameters is essential. For example, in helicopters the plant may vary significantly due to changes in environmental conditions during flight. In applications for space exploration, repair is very costly, and it is preferable to have systems that can adapt to changes caused by aging or the harsh environment of space.

The objective of this research is to derive simple and efficient algorithms for the rejection of periodic disturbances, with an emphasis on time-varying systems. While existing techniques enable one to tackle known systems, few methods are able to deal with time-varying systems and, typically, they assume the existence of a reference sensor.

Of interest in this project are feedback structures that require no reference sensor to feedforward an uncorrupted measure of the disturbance. Already, this problem has been seriously investigated at the University of Utah. We studied several theoretical techniques and found a method that continuously estimates the system dynamics.

By assuming the disturbance is sinusoidal and of known frequency, the number of parameters is much smaller than required to describe the transfer function in ANC systems. From this linear expression, an estimate of both plant and disturbance parameters can be obtained. This estimate can then be used to exactly cancel the disturbance.

The ability to track both rapidly and slowly time-varying systems demonstrates the practicality and robustness of this approach. Similar algorithms can be used for active vibration control and active noise control, and this research is, in particular, validated experimentally on an active noise control test bed at the University of Utah.

Maximally Autonomous Autodirective Antenna Array Technology

75786
R. A. Bates

Project Purpose

Electrically steerable passive array radiator (ESPAR) antennas feature dramatically reduced size, complexity, and power consumption relative to other array types while retaining the ability to achieve high-accuracy beam-forming, direction-of-arrival determination, adaptive tracking, and interference suppression. For example, a typical ESPAR as reported in the open literature uses quarter-wave element spacing, which is one-half of a typical phased array, and achieves electrically steerable gain in the 8-10 dB range over all azimuth angles with the use of only a single receive chain.

Direction-of-arrival accuracy has been reported in the subdegree accuracy levels, and adaptive null formation of better than 25 dB is readily achieved; again all with just the single active receive chain. Although previously verified through prototype measurements, this relatively new technology requires additional development to realize its full potential.

This project, with New Mexico State University, focuses on enhancing the gain, bandwidth, and size advantages of ESPAR technology through enhanced analysis and characterization of a wider variety of array configurations than treated in previously published work, which has focuses almost exclusively on a single, inherently limited array configuration. The analysis approach seeks to come full circle by carefully scrutinizing closed-form approximations of "exact" electric field integral equation (EFIE) formulations of the array system in order to in turn develop reduced-complexity design techniques.

This work has already resulted in one design technique for beam-forming that uses radically simplified computations to directly synthesize array element load values necessary for any azimuthal look angle in arbitrary array configurations. We verified the basic validity of the technique through numerical analysis. Additional work will continue along this paradigm to provide for design techniques that directly treat array bandwidth and gain. The ability to design for both impedance and pattern bandwidth response is especially valuable, and challenging.

Alternate complex analysis configurations, based on using different forms of the EFIE impedance kernel combined with accurate approximations such as the geometric theory of diffraction (GTD), will be used to develop the new reduced-complexity design formulas. Several newer definitions of antenna Q are based on actual element patterns or feed-point impedance trajectories, and will be incorporated into the analysis approach. The results of this work have relevance for all medium-gain, scanned-array applications, and can be used to not only replace larger, more power-hungry existing scanned arrays, but will also allow for scanned arrays to be used in applications where the system cost of such functionality was previously too high.

FY 2006 Accomplishments

We analyzed, simulated, and made measurements with near-earth and subsurface radio frequency (RF) communications. This included analyzing the surface mode traveling waves along the earth-air interface, determining ideal excitation methods for surface waves, and designing hardware for auto-directive antenna arrays and self-matching antenna structures. The results show the potential for overcoming nonideal terrain in diffraction-limited frequency bands, establishing links over non-line-of-sight channels, and decreasing the likelihood of eavesdropping by confining most RF energy, preventing free-space transmission.

We built and characterized a prototype auto-tuning proportional-integral-derivative (PID) feedback circuit. The preliminary results verified the applicability of the approach.

We verified the performance and behavior of a basic ESPAR architecture according to the present state of the art. Design techniques have focused more on initial validation of the most simple ESPAR configuration and application of the architecture to various methods of direction-finding and adaptive beamforming.

We also completed a thorough review of the literature on the topic of ESPAR antennas and completed a summary document detailing the measured performance achieved by various researchers. The document serves as a basis for future research to be performed in this area. We developed a custom two-dimensional method of moments solver to analyze the interelement mutual coupling of an arbitrarily configured ESPAR antenna.

Significance

Many Sandia flight test technologies require advanced antenna structures for satellite and wireless network applications. These applications have impact on advanced concept opportunities for Sandia. Technology developed in this project can benefit intelligence and military applications of network unattended ground sensors and high-gain, electronically steerable antennas for satellite telemetry applications.

The results of this work have relevance for all medium-gain, scanned array applications, and can be used to not only replace larger, more power-hungry existing scanned arrays, but will also allow for scanned arrays to be used in applications where the system cost of such functionality was previously too high.

Generalized Continuum Models for Inelasticity in Solids: Formulation of Theories and Variational Methods for Computation

78783

T. D. Nguyen, K. R. Garikipati

Project Purpose

The purpose of this project is to develop a generalized continuum theory, and numerical implementation of the theory, with embedded length-scales associated with microstructural defects to model inelastic behavior in solids. Classical continuum theories of inelasticity in solids break down when the deformation occurs at scales of a micron and below. In polycrystalline materials, for instance, microstructural features such as grain boundaries, dislocation clusters, microvoids, and microcracks introduce length-scale effects that are not represented in the classical theories.

However, length-scales embedded in additional degrees of freedom associated with generalized continua can be introduced on a physically and mathematically rigorous basis. Therefore, these theories are capable of modeling the influences of microstructure. Such models have gained prominence over the past decade and are being applied actively to model strain localization associated with material softening, large stresses ahead of atomically-sharp cracks, grain size effects, grain rotation and breakage, and the influence of asperities in contact or friction. The most widely-used of such theories introduce length-scales via first and higher gradients of some inelastic strain measure.

There is a large range of these theories, and not all of them are equivalent. Fundamental physical, mathematical and numerical questions remain on the order of strain gradients, the place of higher-order derivatives in governing equations, inelastic strain gradient dependence in yield functions and flow rules, and the application of additional boundary conditions.

In this project with PECASE recipient Krishna Garikapati at the University of Michigan, we are working with a class of generalized continuum inelasticity models of interest to Sandia researchers. The length-scales in these models arise from the density of geometrically necessary dislocations and the disclination density. They are represented by appropriately-defined tensors. The models will be critically-examined in context of the four issues raised above. Recourse will be taken to experimental data in addition to mathematical analysis.

In particular, we will draw heavily from dislocation theory. We will develop vector finite elements and discontinuous Galerkin methods to solve initial and boundary value problems using these models.

FY 2006 Accomplishments

We continued development on vector finite elements (VFE) and discontinuous Galerkin methods for generalized continua. The VFE work was focused on a variant of the BCJ (Bammann–Chiesa–Johnson) plasticity model that includes the effect of plastic incompatibility-based strain gradients. We successfully developed a 2D VFE implementation of this theory and tested its numerical stability and convergence rate.

Our discontinous Galerkin work focused on a strain gradient damage model. We proved stability and convergence and established rates of convergence for both these theories. For the Cahn-Hilliard equation, we established results for time-discrete stability. These accomplishments have laid the groundwork to apply discontinuous Galerkin methods to incompatibility-based strain gradient plasticity theories.

Significance

We focused on developing a novel finite element approach for a variant of the BCJ plasticity model that includes the effect of plastic incompatibility-based strain gradients. The BCJ model is a physically based model for the plastic deformation of metals based on the evolution of microstructural defects such as dislocations and disclinations.

Variants of the model, with different levels of complexity, are currently being used to model the failure of components and systems critical to national security under realistic environmental conditions and situations. Successful completion of this project will result in a robust and more accurate finite element implementation of the BCJ model. Moreover, it will lead to improved numerical methods for modeling other material failure processes such as fracture. This will enhance Sandia's science-based modeling capabilities for safety and reliability.

Refereed Communications

G.N. Wells, E. Kuhl, and K. Garikipati, "A Discontinuous Galerkin Method for the Cahn-Hilliard Equation," *Journal of Computational Physics*, vol. 218, pp. 860-77, November 2006.

Cohesive Zone Modeling of Failure in Geomaterials: Formulation and Implementation of a Strong Discontinuity Model Incorporating the Effect of Slip Speed on Frictional Resistance

79893
J. A. Zimmerman

Project Purpose

Modeling failure in geomaterials due to various loading and environmental conditions is a challenging problem and requires the latest in experimental constitutive modeling and computational solution method technology. Current geomaterial constitutive models and computational methods are incapable of predicting the transition of continuous to fractured geologic material.

As one approach to modeling this transition, in collaboration with graduate student Craig Foster at Stanford University, we developed physically based cohesive zone models for geomaterials and implemented these models within a simulation code that uses the finite element method. This tool advances Sandia's goal of developing a predictive, computational modeling program for failure analysis of geomaterials.

FY 2006 Accomplishments

We developed a new constitutive formulation for modeling the loss of cohesive strength as a material transitions from intact continuum to one with a fully coherent macrocrack. This stage, termed slip weakening, has been coupled with the rate- and state-dependent friction law implemented the previous year, and embedded into the strong discontinuity finite element, also implemented last year.

We tested the numerical implementation to ensure proper convergence of the numerical scheme and to verify that no spurious mesh dependence exists. The element was then used to simulate experimental data to validate its performance. We implemented the element in a way that makes it fully compatible with the available continuum material models; it was tested with Von Mises, Drucker-Prager, and Sandia GeoModel options. Finally, we developed an efficient algorithm and tested it to track multiple localized surfaces throughout a given body.

The results of these simulations were very positive. However, we found that a "kinematic locking" phenomenon, that is partly nonphysical, occurs for curving failure surfaces. This phenomenon can be relieved by allowing the element to open under tensile loading.

Significance

Two problems that would be better understood with this modeling capability are the defeat of hard and deeply buried targets (HDBT) and the long-term performance of deep geologic nuclear waste repositories. It would be useful to be able to predict the behavior of these buried structures when subjected to extreme dynamic loading conditions, such as high-velocity penetration events, explosive blasts, or seismic events.

At present, the mechanics of rock penetration are poorly understood, and there are no empirical data that can be used to forecast long-term performance (over thousands of years) of deep geologic nuclear waste repositories. The advanced model and methods developed in this project overcome limitations of existing methodologies, allowing for more accurate reliability and performance analyses needed for such repositories

In addition to modeling the defeat of HDBT and the long-term performance of nuclear waste repositories, the resulting computational analysis tool will be useful for understanding fracture and fragmentation in geomaterials such as concrete, rock, frozen soil, and heavily overconsolidated clay encountered in fault propagation, tunneling construction, oil and natural gas production, and depleted reservoirs used for subsurface sequestration of greenhouse gases.

Refereed Communications

C.D. Foster, R.A. Regueiro, A.F. Fossum, and R.I. Borja, “Implicit Numerical Integration of a Three-Invariant, Isotropic/Kinematic Hardening Cap Plasticity Model for Geomaterials,” *Computer Methods in Applied Mechanics and Engineering*, vol. 194, pp. 5109-5138, December 2005.

Other Communications

R.A. Regueiro, A.F. Fossum, R.P. Jensen, C.D. Foster, M.T. Manzari, and R.I. Borja, “Computational Modeling of Fracture and Fragmentation in Geomaterials,” Sandia Report, SAND2005-5940, Livermore, CA, 2005.

C.D. Foster, R.I. Borja, and J. Oliver, “Strong Discontinuity Modeling of Slip Weakening and Variable Friction in Geomaterials,” presented at the International Union of Theoretical and Applied Mechanics Seminar on Discretisation Methods for Evolving Discontinuities, Lyon, France, September 2006.

C.D. Foster and R.I. Borja, “Brittle Fracture, Slip Weakening, and Variable Friction Modeling in Geomaterials Using an Embedded Strong Discontinuity Finite Element,” presented at the ALERT Geomaterials Workshop, October 2006.

C.D. Foster and R.I. Borja, “Capturing Slip Weakening and Variable Frictional Response in Localizing Geomaterials Using an Enhanced Strain Finite Element,” in *Proceedings of the Third European Conference on Computational Mechanics*, June 2006.

C.D. Foster, R.I. Borja, and D.D. Pollard, “Continuum Mathematical Modeling of Slip Weakening in Geological Systems,” presented (poster) at the American Geophysical Union Fall Meeting, December 2005.

Reconciling System and Application Logs

80590
S. A. Hurd

Project Purpose

The purpose of this project is to devise a method that can be used to automate the process of mapping events across systems layers in order to assemble them into useful models of events. Such models will aid in both forensic analysis of compromised machines and intrusion detection systems, as they will provide the templates necessary to create useful, easily analyzable logs. This work is in collaboration with the University of California at Davis (UC Davis).

Computer system event logging is best done as a fine balance between overflow of information and lack of data. Limited logging can hinder intrusion detection systems and forensic analysis, both of which rely on the event logs to provide the data necessary to differentiate between normal machine operation and attack. Overly thorough logging, however, can generate so many superfluous entries that the time needed to manually examine the logs for pertinent data is infeasible. Thus, it is critical to devise and maintain a system that can record sufficient data to differentiate between those events that occur in normal operation and those which allude to malicious intent.

A deeper complexity involves the various sources of logs, including both applications and the system (and in many cases, other sources). How to reconcile the log entries generated by the many sources remains an open question in the field. The ability to do so would allow auditors to conserve effort by consulting a collated log, rather than reviewing each source log individually and manually determining which high-layer event corresponds to which low-level entry. Such aggregation is surprisingly complex in today's modern computing environment.

FY 2006 Accomplishments

We found that dealing with clock synchronization issues (both within and between systems) is the critical technical obstacle to project success. The two primary issues are "clock skew" and "clock drift." Without accurate clock synchronization (both within and among systems), it is virtually impossible to effectively perform correlation analysis/reconstruction on logs. Accordingly, we dedicated all resources to solving this problem.

A promising approach to solving this problem was previously developed at UC Davis. This approach, best described as the MIN-DELAY-SYNCHRONIZE algorithm, searches for an entry in the logfile (one for each host), where the difference between the timestamp generated by the host and the timestamp generated by the log-server is minimal. This is assumed to be where the transmission delay was the least and clock skew is estimated using that as a basis.

The MIN-DELAY-SYNCHRONIZE algorithm had only been verified using simulation, rather than using actual systems and associated logs. Thus, the major task we completed this year was to develop and execute an experiment to verify or refute the results of the approach to solving this problem, then analyze the results.

At first, the results we obtained through testing tended to verify the validity of the MIN-DELAY-SYNCHRONIZE algorithm. However, under further examination, it became clear that while this approach works for dealing with clock skew, it does not address issues related to clock drift.

Following this realization, we considered a number of different potential solutions to the clock drift problem. It appears that the most promising approach is to use linear regression techniques in conjunction with the MIN-DELAY-SYNCHRONIZE algorithm. At this time, we have not completed the detailed analysis as to whether this combined approach will work.

Significance

A significant accomplishment from this year is the demonstration that the MIN-DELAY-SYNCHRONIZE algorithm approach is insufficient to deal with both clock skew and clock drift and could lead to flawed research results.

This project supports the national security mission in cyber security by developing improved tools and techniques to use system logs for both intrusion detection and forensics analysis.

Mobile Agent Abstractions, Methods, and Infrastructure for Efficient Sensor Network Tasking Over Heterogeneous Networks

80591
N. M. Berry, L. Szumel

Project Purpose

This research, in collaboration with Ph.D. candidate Leo Szumel at the University of California at Davis (UC Davis), addresses the programming of efficient, scalable, fault-tolerant networks of wireless sensor nodes. These networks are made up of several nodes: elements characterized by a sensor suite, a power source, processing capability, and a radio. There are several challenges in programming these networks.

First, because no single node has complete knowledge of the world environment, nodes of the network must collaborate with each other in order to solve problems with sufficient fidelity and scalability. Second, a network's full potential is only attainable by multiplexing several users or applications, and doing so decreases the effective deployment cost of the network. A third challenge is that limited power means that nodes must spend most of their time sleeping and must perform rapid and efficient collaboration to accommodate that need.

This work integrates the former two challenges into that of "programming," and the latter challenge into that of "communication." This work develops a mobile agent programming model to solve the first challenge and a pheromone-inspired smart flooding communication primitive to solve the second problem.

This approach is to be demonstrated in a real, physical sensor network deployment. The network will be deployed at the UC Davis McLaughlin Reserve to enhance traditional plant ecology studies with new data that would not be attainable via human sampling. The network will measure air, surface, and soil temperatures and soil moisture content.

This deployment has interesting network properties that distinguish it from many experimental networks. First, the network is not in range of wired or wireless data communications. Second, the nodes are placed according to existing experiment sites and cannot be moved to improve radio frequency (RF) connectivity. Third, the terrain and the RF landscape is expected to change considerably as plants grow from foot-high to waist-high. Lastly, a mobile node will visit the site every 1-2 weeks.

This deployment is a specific example of a class of networks that mimics unattended operation in a harsh environment and hybrid static/mobile modalities.

FY 2006 Accomplishments

We examined the efficiency, scalability, and fault-tolerance of sensor network tasks built with the virtual pheromone (VP) primitive, comparing them against state-of-the-art algorithms built using traditional communication methods. VP is found to provide comparable performance while resulting in fewer lines of code.

A high-level network simulator was specifically tailored to the design of agent algorithms and novel communication primitives in sensor networks. The agent high-level pythonic simulator (AHLPS) is based on Python and the SimPy discrete event simulation module. AHLPS allows rapid prototyping and high-level analysis of these algorithms. Implementation in TinyOS and deployment on physical nodes is still used for verification of functionality, but AHLPS provides the unique ability to test algorithms at scale (thousands of nodes) in a reasonable timescale.

At Sandia, we developed and demonstrated a hardware prototype of an adaptive image tracking sensor network (DISCERN). We will continue to work on DISCERN and explore a case study in applying an agent programming model and virtual pheromonecommunication primitive to the DISCERN sensor nodes. This study will provide valuable data and enrich DISCERN capabilities.

Significance

This research focuses on the challenges posed by the programming of large sensor networks. The interest in such networks is quite easy to understand – the more measurements we can collect from our environment, the better we can understand and control it. This argument applies across many fields, including environmental monitoring, physical security, and basic science research.

What has become clear over the last few years, however, is that we are approaching a time when these large networks are affordable from an equipment standpoint but remain a huge logistical challenge, largely because of software development difficulty. These difficulties arise from the need to handle heterogeneous nodes, the spatial and temporal decoupling of nodes, and scaling challenges, among others.

How do we design algorithms that can be tested in simulation and small-scale deployments, then be expected to work well under adverse (perhaps unpredictable) conditions and at a scale that increases over time? How do we amortize infrastructure cost by sharing the network between multiple users? How do we enable adaptation of the network code once it is deployed, in a way that is efficient and does not interfere with existing components that do not need to be updated?

This research is answering these questions by providing a framework for the tasking of, and communication between, sensor nodes in large scale deployments. This framework is scalable, efficient, and can support multiple applications on a single network. The application(s) can be updated independently and/or concurrently.

A specific example of how this research can be applied to Sandia's mission objectives is being demonstrated in the case study. The DISCERN nodes have the ability to detect objects and share their information with neighboring nodes–this is a scalable and efficient design. However, the network is of limited use if all decision synthesis occurs outside the network because doing so limits scalability and increases latency.

We will use the agent framework to program the DISCERN nodes, allowing synthesis to occur inside the network. For example, the user (existing outside the network) can inject an agent that knows how to identify a particular threat and has an action profile dictating how to handle threat detections. The agent then "lives" inside the sensor network, using multi-hop communications only when necessary. A key advantage to this approach is that the learning process can continue long after network deployment; as new threats emerge, new agents can be tailored and injected into the network, at minimal cost, without disturbing the other agents already existing in the network.

Refereed Communications

L. Szumel and J. Owens, "The Virtual Pheromone Communication Primitive" in *Proceedings of the IEEE/ACM International Conference on Distributed Computation in Sensor Systems*, pp. 135-149, June 2006.

Ultrafast Low-Voltage MEMS Switches for Optics and RF Applications

80592
G. N. Nielson

Project Purpose

The objective of this project is to develop and explore a completely new approach to microelectromechanical system (MEMS) switching that has the potential to dramatically increase the speed of MEMS devices while lowering both the voltage and energy required for actuation relative to current MEMS actuation techniques. The new actuation technique utilizes elastic potential energy stored in the MEMS structure to drive the mechanical switching. We are applying this switching technique to the optical domain to create high-speed optical switches.

FY 2006 Accomplishments

In FY 2006, we spent time and effort on increasing the testing capabilities for our devices. We designed and assembled a control circuit that provides both resonant pull-in initialization and strain-energy switching control. We extended the capabilities of the vacuum chamber test setup to allow low-loss transmission of radio frequency (RF) signals into the chamber. We also assembled a test setup to couple light into and out of waveguides on chip for testing waveguide and future switch performance.

We fabricated and tested some parallel plate electrostatic MEMS devices. These devices were tested using the vacuum test setup. This device switched in less than 500 nanoseconds over a switching gap of 1.8 microns with an actuation voltage of 35 volts. This performance is about an order of magnitude better than any other MEMS device with comparable switching gap and actuation voltage.

Using these devices, we also investigated low-stress silicon nitride (silicon rich) and stoichiometric silicon nitride as isolation layers. The silicon-rich silicon nitride seemed to have some propensity for dielectric charging, even in vacuum. The stoichiometric silicon nitride caused the MEMS structures to fracture due to the high intrinsic stress in the film. We investigated the possibility of using high-permittivity dielectrics for isolation in place of the silicon nitride and designed a process to fabricate and test devices with zirconia, hafnia, alumina, and silica isolation layers. We initiated the fabrication of these devices for testing.

We designed, fabricated, and performed preliminary testing of torsional MEMS devices that are precursors to high-speed torsional MEMS mirror devices. These devices are designed to be significantly faster (~ 100X) than the torsional MEMS mirrors that we used last year to initially demonstrate the strain-energy switching and resonant pull-in. Some initial testing of these devices with the newly designed and assembled resonant pull-in circuit indicate that resonant pull-in was achieved at 15 volts, a 25 percent improvement over the quasistatic pull-in voltage that would otherwise be required to pull-in the device.

While strain-energy switching tests are still pending, this device has a resonant frequency of 750 kHz, which should translate into a switching speed of at least 670 nanoseconds. We will continue testing these devices.

In anticipation of combining integrated optics with MEMS devices, we fabricated and tested waveguides to be integrated with MEMS devices to form high-speed integrated optical switches. We fabricated and tested single crystal silicon, polysilicon, and amorphous silicon waveguides. Based on the results of the waveguide investigation, we designed a fabrication process to integrate single crystal silicon waveguides with high-speed MEMS devices.

Significance

Switching is one of the key functionalities that MEMS have provided for a number of domains, including RF, optical, fluidic, thermal, and direct current electrical. Within these domains, MEMS switches are used for a wide variety of switching applications. The dominant technique used for switching is electrostatic switching, although thermal, piezoelectric, and magnetic techniques are also used.

Electrostatic switching has a number of appealing characteristics, such as ease of implementation, low power requirements, and fast switching (relative to the other switching techniques mentioned). One of the primary drawbacks to electrostatic switching is the high "pull-in" voltage required to achieve switching. The MEMS switching technique we are exploring takes advantage of the nonlinear dynamic behavior of electrostatic MEMS systems that allows a significant reduction in the voltage required for switching.

While the primary benefit of this switching approach is a reduction in the voltage required for operation, there are a number of other benefits that result from this technique. The reduction in voltage required allows stiffer structures to be switched at reasonable voltages, which leads to faster switching speeds.

The dynamic behavior is such that the velocity at impact is slower than it would be with the typical electrostatic switching technique where a high voltage is used. The reduction in the impact velocity may lead to improvements in reliability by reducing effects of the initial impact on the microstructure of the material as well as eliminating "bouncing" of the movable electrode on the mechanical stop. Reliability will also be increased as stiffer structures are used because that will help ameliorate the problem of stiction.

Since the device is positively held in both switch states, ringing (i.e., free oscillations) of the switch will be eliminated, which usually extends the switching time significantly. Also, for switching applications in the electrical domain, the positive holding of the device reduces the chance of self-actuation of the device.

Finally, the dynamic switching approach will use significantly less energy for switching. Much of the energy is recycled by converting it from stored potential energy to kinetic energy and then back into stored potential energy, ready to be used again in the next switching operation. The energy saved is reflected in the reduction in the applied voltage. If, for example, the dynamic switch operating voltage is one-quarter of the pull-in voltage, there is a 16X reduction in the energy used.

Since MEMS switching is very widely used in a variety of domains, the impact of these improvements in MEMS switching is significant.

Refereed Communications

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Catalyst Development and Microreformers for Fuel Processing

80593
D. Ingersoll

Project Purpose

The purpose of this project with the New Mexico Institute of Mining and Technology is to develop new supported catalysts for high-efficiency liquid-phase conversion of carbohydrates to hydrogen.

Production of hydrogen (H_2) from biomass and organic wastes is considered an effective approach to mitigating the environmental problems caused by pollutant emissions from fossil fuel combustion and allaying our dependence on the limited oil reserves. However, current technological inefficiencies render such biomass utilization economically unviable. A promising alternative to realizing the goal of hydrogen from renewable sources is to convert biomass-derived carbohydrates to H_2.

Recently, γ-Al_2O_3 supported platinum (Pt/γ-Al_2O_3) catalysts have been successfully demonstrated for the liquid phase reforming of various carbohydrates into hydrogen at temperatures below 300 °C. However, the throughput of the catalytic conversion is low and the cost of the catalyst is high because of the high load of Pt metal, typically in a range of 3 to 5 wt.%. To make the process economically attractive, catalysts with higher performance and lower cost need to be developed.

The main objective of this research is to develop new Pt/NaY zeolite catalysts for high-efficiency liquid (water)-phase conversion of biomass-derived carbohydrates to H_2. The specific tasks of this research include:

- Designing and establishing a packed-bed reactor system for high-pressure liquid-phase reaction
- Synthesizing and characterizing Pt/NaY and Pt/γ-alumina catalysts.
- Selecting catalysts for catalytic performance evaluation.
- Evaluating catalytic performance and stability
- Testing the selected Pt/NaY catalyst for conversion of both ethanol and glucose in liquid phases.

FY 2006 Accomplishments

We synthesized, characterized, and evaluated particulate platinum-loaded Y-type zeolite (Pt/NaY) catalysts for H_2 production from methanol. The catalysts were also tested for liquid-phase reforming of ethanol and glucose to hydrogen. Our accomplishments include:

- We designed and established a packed-bed reactor system for high-pressure liquid-phase reaction. The reactor system was tested by using a similar catalyst (i.e., Pt/γ-alumina) to that reported in the literature.
- We synthesized and characterized Pt/NaY and Pt/γ-alumina catalysts. Synthesis procedures were developed and optimized for both Pt/NaY and Pt/γ-alumina catalysts. X-ray diffraction (XRD) was used to confirm the structure of both catalysts. Microprobe analysis was employed to determine the composition of both catalysts. Scanning transmission electron microscope (STEM) was used to determine the Pt cluster size in the Pt/γ-alumina catalyst. Scanning electron microscope (SEM) was used to determine the size and morphology of the catalyst particles. Brunauer-Emmett-Teller (BET) N_2 adsorption and desorption was used to measure the catalyst surface areas. Carbon monoxide chemisorption was measured to determine the Pt dispersion.
- We selected catalysts for catalytic performance evaluation. Catalysts were selected based on methanol conversion for Pt/γ-alumina and Pt/NaY catalysts with different Pt load levels. The reaction tests were conducted at elevated temperature.
- We evaluated catalytic performance and stability of the selected catalysts for extended periods of operation time (total continuous reaction time more than 200 hours).
- We tested the selected Pt/NaY catalyst for conversion of both ethanol and glucose in liquid phases.

Significance

We developed several methods for preparing nano-platinum supported catalysts showing good catalytic activity. The catalytic performance of the Pt/NaY catalysts have been compared with that of the Pt/γ-Al_2O_3 catalysts reported in the literature as well as those synthesized in this work. The Pt/NaY catalyst with a Pt load of 0.5 wt.% was found to outperform the Pt/γ-Al_2O_3 catalysts with a Pt load of about 3 wt.% for conversion of methanol to H_2.

The Pt/NaY catalyst was demonstrated to be active for ethanol reforming in liquid phase, but incapable of catalyzing glucose because the ringed glucose molecules are too large to effectively transport into the zeolite pores (~ 0.7 nm diameter), where the Pt metal clusters locate. Our research results indicate that the transition metal loaded Y-type zeolite catalysts have great potential for use in catalytic reforming of carbohydrates to H_2.

Other Communications

J. Monroe, "Development of a Platinum-Loaded Y-Type Zeolite Catalysts for High-Efficiency Conversion of Biomass-Derived Carbohydrates to Hydrogen," New Mexico Institute of Mining and Technology Master's Thesis, June 2006.

Catalytic Membrane Development for Microscale Glucose Reforming

80594
D. Ingersoll

Project Purpose

Glucose and ethanol have emerged as the best candidates for biomass derived hydrocarbon processing for hydrogen. Glucose is a major component of biomass; the fermentation of sugars such as glucose produces ethanol, which can then be easily reformed. A fuel-cost analysis indicates that glucose is a more cost effective fuel from any source.

This research with the New Mexico Institute of Mining and Technology examines both the scientific and economic feasibility of microscale glucose aqueous phase reforming for the production of hydrogen. We are conducting experiments to determine optimal conditions for glucose processing. Ethylene glycol is used as a test feed molecule as it contains the same functional groups as glucose and can be processed more expeditiously. We are using the optimal conditions for ethylene glycol reforming in glucose reforming. We are evaluating reactor temperature, pressure, flow rate and fuel concentration for glucose reforming using Pt supported on –325 mesh Al_2O_3 as the catalyst. We are evaluating the surface activity of the catalyst by irreversible CO absorption.

FY 2006 Accomplishments

We evaluated several approaches for immobilizing catalysts on the support. A primary consideration in this regard is effective adhesion of the Pt micro-structured catalyst under the high sheer stress fields associated with the flowing liquid phase in the micro-reactor environment. We used resistance of the catalyst coating to tape adhesion and removal to gauge binding effectiveness.

We developed both electrophoretic deposition and dip coating methods. Electrophoretic deposition is carried out using a Pt-Ru electrode and a stainless steel plate holder with a fixed distance between the two. A potential of 9 V was applied across the electrodes to drive the catalyst to the stainless steel plate in the holder. The dip coating method necessitated development of a mechanized assembly that allowed sample immersion at a rate of 1 mm/minute. Each sample plate remained in the suspension for 1 minute before being removed. After electrophoretic or dip coat deposition of the catalyst suspension, we heat treated each sample plate at 800 °C for 2 hours. Samples prepared in this way show poor adhesion.

Sputter deposition of Pt onto an oxidized stainless steel substrate is also being developed and is expected to produce a sufficiently bonded catalyst coating. Experiments in this area are ongoing. We also integrated a mass spectral residual gas analyzer into the microreactor assembly.

Significance

We completed the microreactor and integrated residual gas analysis system. This will allow characterization of the catalyst structures under conditions of use. Practical advantages of microreactors include safety, “easy modulation,” and numbering up as a means for increasing throughput. Heterogeneous reactions can be carried out efficiently due to short diffusion paths and high surface-to-volume ratios. There are also features that may enable more selective control over chemical synthesis. Reactions run in macroscale batch reactors are usually slow (reaction times of minutes to hours), as fast reactions are difficult to control. The superior mixing and heat and mass transfer in microreactors give the control necessary to carry out fast reactions (reaction times from microseconds to seconds), leading to huge increases in production efficiency.

Heat transfer is one of the more important elements of chemical reaction kinetics. Efficient heat transfer is particularly desirable for fast, highly exothermic reactions. The heat generated by a chemical reaction is proportional to the volume of reagents used, and hence the volume of the reactor. Conversely, heat removal capability decreases with increase in reactor size. Heat produced by the reaction is often removed through the reactor wall, and so the ratio of wall surface area to reactor volume is crucial to efficient heat dissipation. The conduction of heat from highly exothermic reactions and extremely fast reactions in macroscale batch reactors often leads to heat removal as the limiting factor. The high surface-to-volume ratio in microreactors eliminates this problem.

Reliability of Materials in MEMS: Residual Stress and Adhesion in a Micro Power Generation System

80595

N. R. Moody, D. P. Adams

Project Purpose

This project coordinates the work of a Ph.D. student at Washington State University with mentors at Sandia to determine a self-consistent method to quantify interfacial fracture toughness of thin film systems that contain tensile and compressive stresses.

Experimental measurements of adhesion will be coupled with analytical solutions to determine interfacial fracture toughness. These results will provide computational model validation and design parameters and lifetime analysis for micro-electromechanical systems (MEMS) structures and thin films in microelectronics the nation's stockpile.

We used two of the three desired methods of interfacial adhesion, compressed overlayer buckling and four-point bend testing, to measure the interfacial fracture energy of metal-dielectric interfaces. Tested film systems included both Pt films used in MEMS and Au films used in Sandia microelectronics systems. This past year we focused on experimentally characterizing the effect of film chemistry using compressive overlayers and the influence of the applied stress on the calculated toughness.

FY 2006 Accomplishments

We focused on experimentally characterizing the effect of film chemistry on adhesion. We found the adhesion energy of a mixed chemistry interface to be proportional to the coverage of the film with the highest energy interface. This was shown to be important in determining the adhesion energy for nonuniform coverage of interlayers.

In metallic film systems, an interlayer is often used to improve the adhesion. Processing conditions, however, often result in nonuniform coverage. In this set of experiments, the interfacial energy for $Pt/Ti/SiO_2$ initially scaled with nominal thickness of the interlayer and then effectively reached a steady-state value, which suggests a transition from island to uniform film coverage.

We built a four-point bend testing apparatus to measure films at a constant phase angle of 45 degrees. During this test, the film stack of interest is restricted by two elastic substrates on each side and then loaded in a four-point flexure at a constant displacement. With this type of loading, the moment is constant and the energy release rate can be obtained for interfacial cracks in the region between the inner loading spans.

Initial testing of Au and Pt film systems showed that interfacial cracks could be monitored and load plateau could be obtained. These plateaus are proportional to the interfacial fracture energy. In addition, we found that the interfacial fracture energy of film tested with this method needs to have minimum toughness. When the energy is too low, such as in Pt/SiO_2, the interface catastrophically fails and no steady-state crack rate can be measured.

Additionally, we heat treated and analyzed the series of Au films with Cr interlayers using Auger electron spectroscopy to characterize diffusion effects that will impact the toughness measurements. In particular, we identified that Cr acts to improve adhesion both as a complete layer as well as while in solution after heat treated with accelerated aging tests. This suggests that the impact of metallic interlayers on adhesion is a complex phenomenon that will require accurate measurements of both strength and characterization to provide comprehensive data for computational modeling efforts.

Significance

This project is designed to develop self-consistent testing methods and structures for evaluating the reliability of thin films in MEMS and microelectronics with a wide range of properties and stress states. We are producing both experimental methods and analytical models that can provide validation for computational models of interfacial materials properties and design guidelines for MEMS.

The development of testing techniques and analytical models which are rapid, robust, and verifiable will greatly add to the ability of Sandia to evaluate a wide range of critical materials and technologies that currently exist in the stockpile, as well as devices and materials that may exist or be used to monitor these systems in the future. Long-term relationships between Washington State University and Sandia researchers will be enhanced through the campus fellowship. The results from this project are expected to bring a unique capability to Sandia that will enhance our efforts in nanotechnology device design and application.

Refereed

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M.S. Kennedy, A.L. Olson, J.C. Raupp, N.R. Moody, and D.F. Bahr, "Coupling Bulge Testing and Nano-indentation to Characterize Materials Properties of Bulk Micromachined Structures," *Microsystems Technologies Journal*, vol. 11, pp. 298-302, November 2005.

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Modeling River-Aquifer Interaction with Application to the Rio Grande

80596
V. C. Tidwell

Project Purpose

This work addresses a fundamental shortcoming in traditional modeling projects: groundwater and surface water are modeled in essentially different spatial, dimensional, and temporal domains, requiring modelers to focus on either surface or groundwater, with an oversimplified representation of the other. This work focuses on modeling the interaction between surface water and groundwater and its implications for resource management.

Regional groundwater models tend to be three-dimensional (3D), finite element/finite difference models with cell sizes on the order of a kilometer. The temporal resolution is often limited to seasonal or even annual time frames, making support for within-season management decisions difficult. Surface water models tend to represent the river as nodes, which simulate diversions, inflow points, and so on. These nodes are connected by links that roughly simulate the hydrologic behavior of the river between the nodes. Time steps may be days, hours, or even minutes for flow-routing calculations.

The discrepancies between the conceptual representation of surface and groundwater models can be overcome with integrated surface water–groundwater modeling, but the data needs and computational requirements are far greater than for a single domain model. With the rise of advanced code development and geometrically increasing computing power, such model development on a regional scale is now becoming feasible.

This work includes hydrologic modeling of the Rio Grande Project, extending from Caballo Dam in New Mexico to Fort Quitman, Texas, and including diversion of surface water to Mexico at Juarez. Teaming with researchers from New Mexico State University, the Army Corps of Engineers, Bureau of Reclamation, and the Paso del Norte Watershed Council, we will use this data for the formulation and calibration of a coupled hydrologic routing model and groundwater flow model. The resulting model can then be used initially for examining flood routing and ultimately for improving and managing operations of the Rio Grande Project.

FY 2006 Accomplishments

In order to continue development of the Riverware model for the Rio Grande between Caballo Dam and the Rio Grande at El Paso, relationships between the major drain return flows and diversions in the Mesilla Valley reach are needed. We performed analyses to determine if a regression model with autoregressive moving-average (ARMA) errors would adequately describe these relationships. We found that the major drain return flows in this reach were highly correlated to diversions from the river, and we developed ARMA equations for incorporation into the Riverware model

We performed an extensive literature review on stream-aquifer flow interactions. Many subtopics related to this issue were also researched, some of which were: governing equations for flow in open channels; governing equations for ground-water flow; aquifer depletion as a result of well pumping near stream; stream-aquifer interactions in the hyporheic zone; effects of aquifer recharge on stream-aquifer interactions; effects of sediment characteristics at the interface (thickness, heterogeneity, and permeability); effects of bank storage on modeling coupled systems; methods of numerical modeling of the governing equations both as separate systems and as a coupled system; and techniques of software development for object-oriented code modules.

We reviewed and classified a variety of existing models of coupled stream-aquifer systems according to the assumptions that they make with respect to key mechanisms driving the interactions. The primary mechanisms that we identified as possibly being important in stream-aquifer interactions were: the bathymetry of the channel; the sinuosity of the channel; the dimensionality of the equations of state; the distance of the aquifer from the stream; distributed recharge to the aquifer; pumping from the aquifer; aquifer heterogeneity; stream penetration; sediment thickness and heterogeneity; time scales of flow; total versus net water exchange at the interface; and bank storage.

We are developing a Riverware model for the Mesilla Valley portion of the Rio Grande using monthly flow data, and regression models that define the relationships between the diversions and the major drain return flows in this reach. Additionally, we will document the results of the literature review and classifications in a review article. Based on the classifications, the framework for numerical analysis of high-resolution modeling will be developed.

The software coding and baseline analysis to verify and validate the software will also be done. Finally, we will devise a plan that describes the method that will be used to analyze the assumption sets. This plan will start with a system that has few or no assumptions on a hypothetical 3D stream/3D aquifer system with a sinuous stream over a heterogeneous aquifer, and progress to the next simpler assumption set until all assumptions sets have been analyzed.

Significance

Water scarcity has the potential to undermine the nation's economic, energy, and agricultural security. Significant tension exists over water allocations across international and interstate boundaries as well as the distribution of water among irrigators, urban developers and environmentalist. This research will help quantify the magnitude as well as the spatial and temporal variability in exchanges between streams and their adjoining aquifer. For most water courses these exchanges represent a significant contribution to the overall water budget.

Additionally, these exchanges play an important role in defining water quality (including temperature) as well as controlling biologic processes in the hyporheic zone. Unfortunately, it is very difficult to accurately measure these exchanges. Significant difficulties often arise in water planning exercises over uncertainties in surface-groundwater exchanges. To further complicate the water planning process, these uncertainties extend to issues concerning how these exchanges respond to changes in agricultural practices and/or land use within the flood plain.

This research focuses on developing computational tools that better quantify surface water-groundwater exchanges. We compiled an extensive base of literature on the subject that is being synthesized and documented in a comprehensive review article. This article will help organize this knowledge around computational themes and help define the advantages and shortfalls of different schemes according to their underlying assumptions. Once resource gaps are identified, we will develop the next generation of computational tools for modeling river-aquifer interaction. Such models will provide the technical foundation for water resource decision support modeling tools to support of Sandia's goals for water systems.

Kinetics and Mechanisms of Nanowire Synthesis

80598
J. W. Hsu

Project Purpose

In this LDRD project we seek to develop a mechanistic understanding of the heteroepitaxial growth of semiconducting nanowires and nanowire arrays for potential applications in nanoscale electronic, photonic, and sensor devices. Our studies focus on systematically investigating and understanding nanowire growth kinetics of epitaxial silicon and germanium nanowires by ultrahigh vacuum (UHV) chemical vapor deposition (CVD) using the vapor-liquid-solid (VLS) growth technique, such that we can controllably grow specific nanowire heterostructures.

These investigations show that in situ real-time monitoring of nanowire nucleation and growth is essential to understanding nanowire growth kinetics; and as such, we developed an observation method using optical reflectometry to measure nanowire growth in real time. In this technique, treating the growing nanowire layer as an effective dielectric layer and correlating scanning electron microscopy measurements and optical reflection modeling, we show that the technique has surprisingly good sensitivity in observing quantitatively the onset of nanowire growth, the nanowire growth rate, and the average length of the nanowires.

This technique is expected to allow us to accurately control, in situ, the fabrication of nanowire heterostructures. By manipulating the inherent strains within specific Si/Ge heterostructures, we can controllably tune the band structure properties of the nanowires. With Arizona State University, we focus on the key issues, such as the nucleation and growth of the nanowires, the spatially specific location of nanowires into lithographically defined arrays, the factors affecting the synthesis of sharp epitaxial nanowire heterointerfaces, and the resulting strain in these novel structures.

FY 2006 Accomplishments

We continued investigations into the nucleation and growth kinetics of Si and Ge nanowires and Si/Ge heterostructures on Si(111). These studies focus on understanding the fundamental mechanisms of vapor-liquid-solid synthesis of Group IV semiconducting nanowires and exploiting this understanding for the synthesis of novel Si/Ge nanowire heterostructures with new materials properties. We are conducting the research in a specially designed UHV CVD system at Arizona State University.

We developed an optical reflectometry technique for in situ monitoring during VLS nanowire growth, and demonstrated Si/Ge nanowire growth for both linear and core-shell heterostructures. The CVD system has the unique capabilities of in situ metal deposition for the formation of Au catalytic seeds on atomically clean surfaces, and has been extended to provide for millitorr regime growth with up to four precursors (silane, germane, disilane, and digermane) simultaneously.

Our project accomplishments include:

- Developing two new tools for understanding and characterizing nanowire growth: a) first in situ optical reflectometry growth monitoring (using a 635 nm laser) of nanowire growth revealing the presence of an incubation time for nanowire growth, and b) first strain mapping approach for nanowires demonstrating good agreement between experiment and theory for our Si/Ge core shell nanowire heterostructures.
- Using the in situ tools to initiate synthesis studies for axial Si/Ge nanowire heterojunctions, specifically investigating silane and germane in addition to our original disilane and digermane gases; energetic differences in such gases enable specific nanowire heterostructures to be grown. This effort is currently underway. Preliminary

evidence for a growth method to obtain atomically sharp interfaces by thermal cycling of the Au eutectic between liquid and solid states has been revealed.

- Using finite element modeling of the strain in Si/Ge heterostructures along with deformation potential modeling of the resulting band-structure, we developed scaling predictions for core/shell nanowire strain-engineered bandgap tuning for Si and Ge for the first time.

Our results indicate that for [111] oriented Si/Ge core-shell nanowire heterostructures it will be possible to "tune" the bandgap of Ge cores or shells by up to a factor of two through the ratio of core-to-shell thicknesses. This unprecedented level of bandgap modification results from the unusually large strains possible in nanowire heterostructures and the fact that the conduction band minimum for Ge is determined by the L direction, which is particularly sensitive to strain along the [111] direction. In contrast, the Si bandgap is only modified by a few 10s of meV. This contrast points to the importance of controlling the nanowire orientation in conjunction with nanowire heterostructure design.

The tuning of the bandgap opens the door to increased bandstructure control and novel electronic properties, such as one-dimensional hole gas structures. In addition, we initiated a collaboration to grow nanowire arrays using Au seeds patterned by electron beam lithographically. This approach is the first step to exploiting heterostructure designs to fabricate advanced nanodevices.

Significance

A key research and development accomplishment of this project is the determination that the electronic band structure of semiconducting nanowires can be tuned over a significant range of properties. Large modifications in the electronic band structure can be achieved through the growth of nanowire core-shell and axial heterostructures using conventional electronic materials such as silicon and germanium. The changes in bandstructure are predicted to make possible electronic and optoelectronic properties not previously attainable. These modifications in electronic properties are due to the large strains that can be incorporated without the introduction of strain-relieving defects, something which cannot be achieved in normal semiconductor processing such as conventional CVD or MBE layered growth.

While additional work is needed to clearly demonstrate the various aspects of this important result, it is apparent that new electronic properties will be achievable in nanowire electronics. Potential implications and future applications of this result include room temperature resonant tunneling diodes made from Si/Ge, nanowire logic circuits at ultrasmall dimensions, ultrasensitive chemical sensor arrays, and high-performance thermoelectric devices. Such advances would have beneficial impact on national security and energy utilization missions.

Refereed Communications

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Rapid Chemical Analysis Using Micropower Gas Chromatographic Columns and Latching Microvalves

80599
A. Robinson

Project Purpose

Chemical sensing is critical for a wide variety of applications, including those in industrial process control, environmental monitoring, health diagnosis, and homeland security. Individual sensors often lack speed, sensitivity, stability, and (especially) selectivity. Gas chromatography (GC) provides a solution to these problems, but commercial instruments are bulky, power-hungry, and inefficient at best. Thus, this project seeks to develop GC components for a high-performance separation and detection, ultraminiature (1 cubic centimeter), micropower gas chromatography system capable of analyzing a wide variety of gaseous mixtures in seconds with high selectivity and part-per-billion sensitivity. We are also investigating electrostatically latching thermopneumatic micro-valves for the purpose of pressure and flow tuning and programming the separation. This project is in collaboration with the University of Michigan.

FY 2006 Accomplishments

The microfabricated GC column designed in this project has substantially smaller size and thermal mass, and higher chromatographic performance than any reported in the past. The goal is to perform separations at temperatures above 100 °C, using less than 20 milliwatts of power. A thermal ramp rate on the order of seconds is sought, with the ability to separate 30+ compounds in less than 5 minutes.

Initially, we used a simple column fabrication process for rapid device design and testing. This process utilized a deep reactive-ion etch (DRIE) followed by anodic bonding of a glass plate to close the channel. Columns were coated with polar and nonpolar stationary phases to achieve separations of chemical compounds. The nonpolar, 3-meter column has separated over 30 components in less than 6 minutes, achieving over 4000 theoretical plates per meter. These characteristics facilitated miniaturization, but posed significant design challenges. We incorporated integrated pressure sensors with thin boron-doped silicon membranes. The pressure sensors are necessary for repeatability in measurements, as well as for failure detection in the column.

A second approach to column formation utilized a buried microchannel that we sealed through chemical vapor deposition and then etched back to remove the unnecessary substrate. The column was fully suspended to reduce heating power consumption and to improve its thermal ramping rate. The device requires only 11 milliwatts to maintain the 25-centimeter long column at 130 °C. This low-mass, isolated column requires five times less power and is four times faster (thermally) than any other column to date. Furthermore, it achieves over a five-fold improvement in separation performance over previously reported low-power columns.

In order to achieve the aggressive valve goals, we are developing and improving thermopneumatic valves for low power and fast operation. The approach utilizes a thermally isolated thermopneumatic chamber and an electrostatic latch, harnessing the advantages of both actuation mechanisms. Initial testing for the fabricated microvalves revealed an open flow rate of 8 standard cubic centimeters per minute at a differential pressure of 5 torr, and a leak rate of 0.001 standard cubic centimeters per minute at 860 torr.

The thermopneumatic actuator closes the valve in under 430 milliseconds with 250 milliwatts. In conjunction with the electrostatic latch, 60 milliwatts is required to keep the valve closed. The integrated valve plate position sensor has a sensitivity of 1.3 femtofarads per torr and is used to monitor when the valve has closed and, thus, when to activate the electrostatic latch.

Significance

Gas chromatography is one of several highly versatile, highly sensitive chemical analysis methods. It is the most amenable to extreme miniaturization. The research and advances made in this project serve to retain the adequate analytical fidelity of the method as further miniaturization occurs. At present, GC has been miniaturized to a battery-powered hand-held instrument. This is suitable for military and first responders to perform air monitoring for toxic chemicals and chemical warfare agents.

To use this instrument, a technician must pause from activities, use a keypad, and wait at least one minute (two minute run-to-run cycle time). Batteries, creating the heaviest mass of the entire system, last approximately four hours. Extreme miniaturization and commensurate savings in power consumption will lead to micro-GC systems weighing ounces that can run autonomously for days unobtrusively in a package similar to a button. This also opens the door for surveillance using unmanned aerial vehicles, distributed ground sensors, and other leave-behind methods.

Because GC is such a versatile technique micro-GCs will find use in gas refineries, pharmaceutical manufactures, chemical factories, semiconductor manufacture facilities, etc. GC breath analysis for medical diagnosis is presently under study. These and other applications give this research project impact in energy, as well as military, nonproliferation, and homeland security missions.

Refereed Communications

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C.J. Lu, W.H. Steinecker, W.C. Tian, M.C. Oborny, J.M. Nichols, M. Agah, J.A. Potkay, H.K.L. Chan, J. Driscoll, R.D. Sacks, K.D. Wise, S.W. Pang, and E.T. Zellers, "First-Generation Hybrid MEMS Gas Chromatograph," *Lab on a Chip*, vol. 5, pp. 1123-1131, October 2005.

Other Communications

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Automated Assembly of Microscale Devices

80601
J. F. Jones

Project Purpose

Microelectromechanical systems (MEMS), with their ability to integrate moving mechanical parts, electronics, and other functions inexpensively, have had a deep impact on many fields of endeavor. We are just beginning to see their amazing potential to provide tangible benefits for our daily lives. On the other hand, MEMS and their related technologies are drastically limited in several important ways.

The key problem is the inability to form true three-dimensional (3D) structures of the needed complexity comprised of heterogeneous materials. Without solutions to this problem, the range of future applications will be severely restricted. If effective solutions can be found, they will revolutionize the way in which numerous products based on microscale parts will be produced. We are collaborating with Carnegie Mellon University (CMU) to address the main robotic issues regarding the assembly of complex 3D microscale mechanisms and the integration of different microfabrication technologies into hybrid microsystems.

The approach is aimed at the system level, building on previously developed MEMS batch and robotic assembly methods. Unlike previous work, we are applying a robotic-agent-based distributed information architecture for rapidly deployable and rapidly reconfigurable microassembly systems. In contrast to previous robotic systems for microassembly based on individual work cells, this approach uses a modular branching pipeline concept to address scalability in four main dimensions with the same computational and physical system. These dimensions are: product size scalability, fastening process scalability, parallel processing scalability, and assembly process step scalability.

Ultimately, this research will provide the basis for modular and scalable microfactories that will enable the economically viable assembly of complex 3D microscale mechanisms and integration of hybrid microsystems relevant to NNSA nuclear weapons applications.

FY 2006 Accomplishments

We developed a new gripper concept for integration into the CMU minifactory environment. We fabricated and functionally tested the new gripper concept. Small grippers such as MEMS-based microgrippers tend to be fragile and easily damaged. Sandia has shown that commercially available nonmagnetic tweezers can be used successfully to pick and place objects such as metal spheres as small as 30 µm in diameter. A significant advantage is the fact that a tweezer with bent or misaligned tips can be easily replaced.

The Sandia system uses a small model airplane servo and linkage to actuate the tweezers. CMU adopted this approach for the new microscope end effectors. In contrast to the prior approach, the CMU system operates the tweezers by a small voice coil actuator. The actuator's magnet assembly is attached to one arm of the tweezers and the voice coil is attached to the other. This enables the tweezer arms to move symmetrically at the center of the microscope's field without any friction. Additionally, the tweezer gripping force is simply proportional to coil current, making control straightforward

Significance

The evolution of 3D heterogeneous microsystems holds promise to enable a range of revolutionary capabilities of interest to national security. These capabilities range from ultraminiature weapon components to highly covert sensor systems. However, the assembly of 3D microscale mechanisms and integration of hybrid microsystems present significant technical challenges. This research contributes to moving toward a practical manufacturing basis for realizing assembled microsystems.

MEMS Reconfigurable Intelligent RF Circuits

80602
C. W. Dyck, C. D. Nordquist, G. M. Kraus

Project Purpose

Switched impedance networks are essential in developing reconfigurable systems, however "tuner" networks are more complex and produce many points over a targeted impedance range. Such networks could be used as chip-level load-pull networks in order to increase the efficiency of high-power systems, reduce design cycle times, and enable dynamic system optimization. Conventional power amplifier design uses mechanical source and load tuners to determine the optimal operating impedances for highest power and efficiency. However, high-power applications often require several amplification stages, and there exists no method to determine the optimal interstage impedance in these multistage amplifiers.

A MEMS (microelectromechanical system) tuner network could be placed between each amplifier stage and used to vary the impedance for optimal power and efficiency. These networks could either remain in the amplifier to allow it to be dynamically optimized with changing signal and environmental conditions, or removed and used solely as a measurement tool.

FY 2006 Accomplishments

We designed and fabricated a novel design of an impedance transform network at the University of Colorado at Boulder. The design was an electrically reconfigurable double-slug network consisting of 22 RF MEMS ohmic switches. We designed this network to cover large portions of the Smith chart while dramatically improving on the loss of previous impedance transform network topologies. The system was designed using Agilent's Advanced Design System (ADS). Masks were ordered and the circuits were fabricated at Sandia using the baseline RF MEMS ohmic switch process. Testing was not completed.

Significance

This development of reconfigurable active networks benefits the DOE by developing a knowledge base and infrastructure at Sandia to address national security and nuclear weapon needs in the area of high-performance miniaturized microwave systems. Potential applications in communications, radar, and surveillance include switched-mode operation, frequency hopping for antijamming, and dynamic adaptive reconfiguration in response to environmental changes.

Patrick Bell completed his PhD thesis on MEMS reconfigurable networks while supported by this project, and graduated from the University of Colorado at Boulder during FY 2006.

Other Communications

P.J. Bell, "Reconfigurable Microwave Power Amplifiers," PhD dissertation, University of Colorado at Boulder, 2006.

Bayesian Inference for Inverse Problems, Model Structure, and Uncertainties

80603
Y. Marzouk, H. N. Najm, L. A. Rahn

Project Purpose

Inverse problems are of great relevance to science and engineering. While progress in detailed, first-principles modeling of physical systems has been enormous, this progress has exposed a challenging and complementary task – inferring unknown model parameters, model inputs, and model structures from data in realistic problems.

Several factors make these inference problems difficult to solve. Real-world observations are often sparse and are inevitably affected by noise and measurement error. Inversion is typically ill-conditioned; small errors in measurement can lead to enormous changes in the estimated model or model parameters. Moreover, multiple models may match a given data set, or no model may match the data.

This work develops a probabilistic setting for inverse problems, based on Bayesian inference. The Bayesian formulation provides a rigorous foundation for inference from parsimonious and noisy data, a natural mechanism for incorporating disparate prior sources of information, and a quantitative assessment of uncertainty in the inferred results. We also seek to develop efficient computational tools that surmount the cost of evaluating high-dimensional Bayesian integrals or probing posterior distributions, particularly for complex forward problems accessible only through expensive detailed simulations.

We consider two key applications: contaminant source inversion and gene regulatory network construction. Progress in algorithms for source inversion is important to homeland security, in response to current risks of bioagent or toxin release in public spaces. Efficient Bayesian inference from sparse and noisy data will provide well-founded estimates of confidence in threat characterizations, enabling better targeted and more effective responses. Robust inference of gene regulatory network structure may fundamentally enhance knowledge of crucial aspects of cellular function, from elucidating pathways between normal and diseased cellular states to clarifying and manipulating responses to pathogens and toxins. This work will thus impact Sandia's portfolio in biotechnology and molecular biology. Principal investigator (PI) Youssef Marzouk is a Truman Fellowship Award recipient.

FY 2006 Accomplishments

Work in FY 2006 focused on stochastic spectral reformulations of the Bayesian approach to inverse problems. We first completed the theoretical foundation for these new approaches, then explored the accuracy and efficiency of the resulting methods by demonstrating them in practical applications.

In time-dependent contaminant source inversion problems, we demonstrated that using polynomial chaos (PC) expansions to propagate a wide range of uncertainty, e.g., prior uncertainty, through the forward problem, and sampling the resulting spectral expansion, enables a substantially more efficient Bayesian solution of the inverse problem. This new formulation addresses a core computational challenge associated with the Bayesian formulation – the cost of evaluating high-dimensional integrals over the posterior, particularly when each sample or quadrature point requires solution of an expensive forward problem.

Next, we focused on the inference of continuous spatial-temporal fields. This class of problems includes source inversion where the source field is allowed to have an arbitrary space-time dependence; it also includes material property inversion, such as estimating the permeability or diffusivity of a material from sparse and/or remote measurements of scalar transport. Solving these problems in a Bayesian context required developing new methods for dimensionality reduction. We appealed to the

Karhunen-Loève (KL) expansion of stochastic processes, essentially using the prior to develop basis functions for the unknown field. This converts the full-dimensional inference problem to Markov chain Monte Carlo (MCMC) exploration of the joint posterior distribution of the KL mode strengths. Results on transient problems with spatially varying diffusivity show excellent agreement with the full-dimensional formulation and substantial speedup. Moreover, they allow polynomial chaos acceleration of Bayesian inversion in high-dimensional problems.

We implemented all of this work in a flexible and extensible code framework.

Significance

The first focus of this work, new algorithmic developments for Bayesian inference in complex inverse problems, should have a growing impact on both the inverse problems and the statistics communities. By introducing efficient spectral methods for uncertainty propagation into the realm of inverse problems, we are making Bayesian inference with detailed physical models far more computationally tractable. We expect that this will facilitate the adoption of Bayesian approaches, with their rigorous treatment of data noise and model uncertainty, by inverse problems practitioners. We also hope that these techniques will enable statisticians to employ more realistic, first-principles, physical models when analyzing data.

Impact on specific application areas begins with the prototypical source inversion problems we used to demonstrate our formulation. Contaminant source inversion is at the heart of pressing problems in homeland security, i.e., responding to bioagent or toxin releases in public spaces. Efficient Bayesian inference from sparse and noisy data will provide well-founded estimates of confidence in threat characterizations, enabling better targeted and more effective responses. Applications of inverse problems in Sandia's mission areas extend much further. Geophysical applications, e.g., characterizing subsurface properties from remote and indirect measurements, are fertile ground for considering uncertainties in physical models, measurements, and the resulting inverse solutions. We hope to leverage our work on Bayesian inference for continuous-field inverse problems into this area.

Experience with computational tools for Bayesian inference, developed in this project, has already begun to influence several other Sandia efforts, including:

- "Practical Reliability and Uncertainty Quantification for Complex Hierarchical Systems," LDRD Project 105814. Starting in FY 2007, this new project will use Bayesian techniques to estimate and extrapolate the reliability of complex systems from test data, with quantifiable credibility bounds.
- "Distributed Microreleases of Bioterror Pathogens: Threat Characterization and Epidemiology from Uncertain Patient Observables," LDRD project 93505. Started in FY 2006, this project uses Bayesian inference strategies to characterize an unfolding bioterror attack based solely on scarce patient data, in the earliest phases of an outbreak.
- A BES-funded Sandia project on analysis of experimental data in combustion chemistry, using Bayesian analysis and computational tools (e.g., MCMC) to construct uncertain chemical models.

Refereed Communications

Y.M. Marzouk, H.N. Najm, and L.A. Rahn, "Stochastic Spectral Methods for Efficient Bayesian Solution of Inverse Problems," in *Proceedings of the Bayesian Inference and Maximum Entropy Methods in Science and Engineering*, pp. 104-110, August 2005.

J. Ray, Y.M. Marzouk, H.N. Najm, M. Kraus, and P. Fast, "Estimating Bioterror Attacks from Patient Data: A Bayesian Approach," submitted to *Proceedings of the RAND/ASA Conference on Quantitative Methods & Statistical Applications in Defense and National Security*.

Y.M. Marzouk, H.N. Najm, and L.A. Rahn, "Stochastic Spectral Methods for Efficient Bayesian Solution of Inverse Problems, *in Proceedings of American Institute of Physics,* pp. 104-110, February 2006.

Other Communications

H.N. Najm, T.B. Settersten, and Y.M. Marzouk, "Uncertainty Estimation from Experimental Data: Bayesian Analysis of NO Fluorescence Quenching," presented at the 31st International Symposium on Combustion, Heidelberg, Germany, August 2006.

Y.M. Marzouk, "Stochastic Spectral Methods to Accelerate Bayesian Solution of Inverse Problems," presented (invited) at LANL statistics seminar, Los Alamos, NM, June 2006.

Y.M. Marzouk and H.N. Najm, "Stochastic Spectral Methods for Efficient Bayesian Solution of Inverse Problems," presented at the 3rd World Conference on Computational Statistics and Data Analysis, Limassol, Cyprus, October 2005.

Y.M. Marzouk and H.N. Najm, "Polynomial Chaos Acceleration of Bayesian Methods for Inverse Problems," presented at the SIAM Conference on Parallel Processing for Scientific Computing, San Francisco, CA, February 2006.

Y.M. Marzouk and H.N. Najm, "Stochastic Spectral Methods to Accelerate Bayesian Solution of Inverse Problems," presented at the Valencia/ISBA 8th International Meeting on Bayesian Statistics, Benidorm, Spain, June 2006.

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Atomistic Modeling of Nanowires, Small-Scale Fatigue Damage in Cast Magnesium, and Materials for MEMS

80604
J. A. Zimmerman

Project Purpose

Lightweight and miniaturized weapon systems are driving the use of new materials in design such as microscale materials and ultralow-density metallic materials. Reliable design of future weapon components and systems demands a thorough understanding of the deformation modes in these materials that comprise the components and a robust methodology to predict their performance during service or storage.

Traditional continuum models of material deformation and failure are not easily extended to these new materials unless microstructural characteristics are included in the formulation. For example, in LIGA (for the German term *Lithographie, Galvanformung, und Abformung,* for lithography, electroforming, and molding) Ni and Al-Si thin films, the physical size is on the order of microns, a scale approaching key microstructural features. For a new potential structural material, cast Mg offers a high stiffness-to-weight ratio, but the microstructural heterogeneity at various scales requires a structure-property continuum model. Processes occurring at the nanoscale and microscale develop certain structures that drive material behavior.

The purpose of this work, with PECASE recipient David Miller of the University of Colorado at Boulder, was to understand material characteristics in relation to mechanical properties at the nanoscale and microscale in these promising new material systems. Research was conducted to employ tightly coupled experimentation and simulation to study damage at various material size scales under monotonic and cyclic loading conditions.

Experimental characterization of nano/microdamage was accomplished by novel techniques such as in situ environmental scanning electron microscopy, 1 MeV transmission electron microscopy, and atomic force microscopy. New simulations to support experimental efforts include modified embedded atom method (MEAM) atomistic simulations at the nanoscale and single crystal micromechanical finite element simulations.

FY 2006 Accomplishments

We focused on three topics:

Atomistic Simulations of Metallic Nanowires

We focused on the plastic properties of the nanowires and examined the effects of wire size, orientation, and structure on yielding of nanowires in a systematic manner. We examined slip, twinning, and reversibility in metal nanowires, and made a major discovery on shape memory and pseudoelasticity in metal nanowires.

Properties of Thin Films for Microelectromechanical Systems (MEMS)

We focused on thin Au film on Si microcantilevers, corrosion of Si structures, and material properties from SiO MUMPS™ designs. We began testing SiO MUMPS parts and the development of a miniature tensile frame.

Major findings include the ability of corrosion to influence device properties in the presence of a metal layer, evolution and intrinsic stress development in Au/Si thin films, and the mechanical properties of SIO materials.

Nanoindentation of NiTi Shape Memory Alloys

Nickel-titanium (NiTi) shape memory alloys undergo relatively large recoverable inelastic deformations via a stress-induced martensitic phase transformation. Although stress-induced phase transformations in shape memory alloys are well characterized and used at micrometer to meter length-scales, significant opportunity exists to understand and exploit martensitic transformations at nanometer scales.

Displacive stress-induced martensitic phase transformations may constitute an ideal nanometer-scale actuator, as evident in certain biological systems, such as the T4 bacteriophage. We used nanoindentation to study the fundamentals of stress-induced martensitic phase transformations in NiTi shape memory alloys. Results showed evidence of discrete forward and reverse stress-induced thermoelastic martensitic transformations in nanometer-scaled volumes of material.

We demonstrated shape recovery due to indentation, followed by subsequent heating, for indent depths in the sub-10 nm range. The indentation results revealed that stress-induced martensitic phase transformations nucleate at relatively low stresses at nanometer scales, suggesting a fundamental departure from traditional size scale effects observed in metals deforming by dislocation plasticity.

Our results revealed that the local material structure can be used to modify transformation behavior at nanometer scales, yielding an insight into the nature of stress-induced martensitic phase transformations at small scales and providing an opportunity for the design of nanometer-sized NiTi actuators.

Significance

The project created a large database of both experimental and computational results that link characteristics such as structure and defect content to a material's mechanical properties. This increase in knowledge is a huge boon as the trend in engineering systems has been to, and will continue to, take advantage of nanoscale and microscale structure to achieve desired materials properties.

In addition, the methods we employed can be applied to other materials of interest to existing Sandia technology, as well as future devices and technologies that would be of interest for Sandia mission needs. For example, the computational models and methods used to examine deformation and failure of nanowires can and will be applied to other nanowire materials, as well as nanoscale structures of other shapes, e.g., thin films, as the use of these materials becomes more commonplace in Sandia technology and the need for a predictive simulation tool becomes paramount.

Refereed Communications

J. Diao, K. Gall, M.L. Dunn, and J.A. Zimmerman, "Atomistic Simulations of the Yielding of Gold Nanowires," *Acta Materialia,* vol. 54, pp. 643-653, February 2006.

H.S. Park, K. Gall, and J.A. Zimmerman, "Shape Memory and Pseudoelasticity in Metal Nanowires," *Physical Review Letters*, vol. 95, pp. 255504/1-4, December 2005.

M.K. Tripp, C. Stampfer, D.C. Miller, T. Helbling, C.F. Herrmann, C. Hierold, K. Gall, S.M. George, and V.M. Bright, "The Mechanical Properties of Atomic Layer Deposited Alumina for Use in Micro- and Nano-Electromechanical Systems," *Sensors and Actuators A*, vol. 130, pp. 419-429, August 2006.

D.C. Miller, K. Gall, and C.R. Stoldt, "Galvanic Corrosion of Miniaturized Polysilicon Structures: Morphological, Electrical, and Mechanical Effects," *Electrochemical and Solid State Letters*, vol. 8, pp. G223-G226, 2005.

D.C. Miller, W.L. Hughes, Z.L. Wang, K. Gall, and C.R. Stoldt, "Mechanical Effects of Galvanic Corrosion on Structural Polysilicon," to be published in the *Journal of MicroElectroMechanical Systems*.

H.S. Park, K. Gall, and J.A. Zimmerman, "Deformation of FCC Nanowires by Twinning and Slip," *Journal of the Mechanics and Physics of Solids*, vol. 54, pp. 1862-1881, September 2006.

D.C. Miller, M.J. Talmage, and K. Gall, "Incipient Yielding Behavior During Indentation for Gold Thin Films Before and After Annealing," to be published in the *Journal of Materials Research*.

C.P. Frick, T.W. Lang, K. Spark, K. Gall, "Stress-Induced Martensitic Transformations and Shape Memory at Nanometer Scales," *Acta Materialia*, vol. 54, pp. 2223-2234, May 2006.

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D. Miller, C.F. Hermann, H.J. Maier, S.M. George, C. Stoldt, and K. Gall, "Thermomechanical Stability of Thin Film Multilayers: Part II Microstructure Evolution," to be published in *Thin Solid Films*.

Design, Analysis and Control of MEMS Devices for Micromanipulation Tasks

80667

J. J. Allen

Project Purpose

The objective of this research is to develop advanced microelectromechanical systems (MEMS) micromirror arrays for use in adaptive optics applications such as optical communications and imaging systems. In order to realize the full capabilities of MEMS optical systems for the demands of next-generation technologies, MEMS micromirror components need to be robust and agile to the changing needs of performance and environment with precise and accurate positioning. Micromirror arrays are one of the most successful and versatile MEMS applications, including optical switches for telecommunications, scanning and imaging for projection displays, diffraction gratings for spectroscopy, and adaptive optics for wave front correction.

Many of these devices require large arrays of micromirrors and it is desirable to ensure accurate positioning capabilities for each mirror in the array despite the presence of outside disturbances or variations from the fabrication process such as small deviations in dimensional or material properties across the array. Many of today's emerging technologies require true analog positioning capabilities and in order to guarantee precision and accuracy of the mirror position for analog operation, closed-loop feedback control techniques are needed. Feedback control has long been used in many macroscale systems to correct for such factors, yet limited work has been done to apply these techniques to MEMS systems.

Current state-of-the-art micromirror arrays either rely on discrete open-loop actuation that may limit the device to on/off binary operation or require extensive calibration to simulate analog performance. For applications that require continuous analog positioning of very large arrays of mirrors (on the order of millions), these are not optimal or efficient solutions. The device performance must be robust with respect to parametric uncertainties from the fabrication process as well as environmental noise and uncertainties in the feedback-loop from the sensor.

This research is focused on the development of micromirrors with closed-loop control to ensure accurate position tracking across an array of devices in the presence of parametric uncertainties and external disturbances. This goal will be achieved by considering both optimized design of the actuators as well as developing closed-loop control schemes to meet the unique needs of MEMS systems. The success of this project will lead to new capabilities for MEMS optical systems and provide means for increased reliability and precision.

FY 2006 Accomplishments

We filed for a patent on a mechanism design that seeks to eliminate the problem of electrostatic pull-in on the micromirrors. Pull-in is a common feature of electrostatic actuators and limits the range of analog motion of the mirror and also creates hysteresis in the motion of the device. Eliminating this problem will ensure greater range of motion and stability for the micromirror arrays.

We completed designs of new micromirror arrays that incorporate on-chip piezoresistive position sensing. The new designs have been fabricated in SUMMiT V™ and await further testing. The designs will establish the capabilities of creating piezoresistive sensing within the standard SUMMiT process by utilizing the process layers in a novel fashion. In addition the new arrays will be used to study the effectiveness of using either distributed (i.e., every micromirror's position is sensed) or localized (i.e., only a few mirrors' positions are known) sensing to achieve active control of the entire micromirror array.

We continued development of the device model for use in design and simulation of the closed-loop control system and identified expected sources and values of uncertainties to incorporate into the model for robust control design. We developed an optical testing method to obtain real-time data and create a test bed for experimentation.

Significance

The full impact of microsystem technology has thus far been limited by a lack of reliable, accurate, and high-precision MEMS devices. This project will lead to new capabilities for MEMS optical systems and provide a means for strengthening Sandia's national security capabilities by integrating more reliable and precise microsystems into next-generation communication and imaging technologies.

Our accomplishments have provided the fundamental modeling as well as the design and fabrication of an experimental module that will enable viable demonstration of passive and active control methodologies. The objective is to achieve micromirror arrays with precise and accurate positioning enabled by use of robust, optimized design and control techniques.

The successful completion of this work will allow micromirrors for adaptive optics applications that are robust to parametric uncertainties that commonly arise through microfabrication processes as well as to disturbance rejection and plant instabilities. The application of optimal design methods and closed-loop control techniques will enable cost reduction as the devices will no longer require extensive calibration for open-loop performance, as well as improved performance and reliability. Further, the incorporation of on-chip sensing mechanisms into the device will allow for compact realization of complete microsystems. This method for using piezoresistive methods within SUMMiT V fabrication is novel and its success will open up new areas of device applications.

Other Communications

J.R. Bronson, G.J. Wiens, and J.J. Allen, "Modeling and Alleviating Instability in a MEMS Vertical Comb Drive Using a Progressive Linkage," in *Proceedings of the ASME International Design Engineering Technical Conference*, September 2005.

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Developing Novel Scaffolds for Biological Molecules by Solving the I-QSAR Problem Using the Signature Molecular Descriptor

82854
J. M. Faulon

Project Purpose

This project is aimed at designing molecular compounds having specific biological activities and properties. This goal is accomplished in two steps. One creates a quantitative structure activity relationship (QSAR) between a set of known compound structures and their experimentally measured activities. In this first step, named forward QSAR, molecular structures are described using the signature molecular descriptor, which has previously been shown to give good accuracies in property and activity predictions.

The second step, named inverse-QSAR, consists of finding the structural elements (i.e., the signatures) that best match a given target activity and enumerating all the compounds matching these structural elements. This step comprises a set of Diophantine equations that are solved to find the signatures that match the targeted activities, and an algorithm that enumerates all molecular structures corresponding to a given signature.

In FY 2006, we benchmarked the technique and used it to design protein inhibitors as well as environmentally friendly foam-blowing compounds.

This work is being performed in collaboration with PECASE recipient Donald P. Visco Jr. of Tennessee Technological University.

FY 2006 Accomplishments

We generated knowledge about what constitutes complicated inverse design problems using the signature molecular descriptor and potential solutions to the various bottlenecks that arise. We used template systems to develop inhibitors for g-secretase as well as inhibitors of Cdc25.

By working with these challenging systems (molecule size 50 – 100 atoms), we experienced a myriad of both computational and size-related issues, which forced us to refine the general inverse design algorithm. In particular, we will use a lower-signature height to generate a large pool of potential solutions (coarse-grained) and use a higher-signature height to refine that pool in order to identify the best candidate solutions to the problem.

Within this larger algorithm, we developed a modified brute-force solution technique to solve the system of Diophantine equations that result in the inverse design process with signature. This modified solution technique allows us to estimate the computational complexity of the problem and estimate the time required for solution using both the space and CPUs (central processing units) at hand. When problems become intractable, then larger, more robust approaches must be initiated.

Other work we accomplished:

- Incorporated multiple steps via multiple codes in the inverse solution algorithm into one, large script
- Generated post-processing scripts to filter nonphysical structures from structure generation code
- Developed parallel code to more efficiently implement the modified brute-force approach to solve the Diophantine equations.

Significance

The concept of designing compounds matching targeted properties and activities is novel and has not been successfully developed and applied prior to this work. We have filed a patent on the methodology developed in this project. While there are still bottlenecks we need to overcome, in particular to design compounds of high molecular weights (> 50 nonhydrogen atoms), our technique can and has been used to design novel compounds.

There are many potential applications for this work, ranging from the design of novel materials having better physical properties (polymers for instance), to the design of environmentally friendly chemicals (foam-blowing agents), and the design of drugs that are more potent to a specific target (inhibitors of g-secretase, Cdc25, and cox-2).

Refereed Communications

D. Weis, J.L. Faulon, R.C. LeBorne, and D.P. Visco, "The Signature Molecular Descriptor 5: The Design of Hydrofluoroether Foam Blowing Agents Using Inverse-QSAR," *Industrial & Engineering Chemistry Research*, vol. 44, pp. 8883-8891, November 2005.

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File System Performance Optimization for Supercomputing Applications

85512

R. A. Oldfield

Project Purpose

Scalability demands on the file systems of future supercomputers are giving rise to new system-level technologies. The purpose of this work with the Georgia Institute of Technology is to explore and experiment with ways in which applications interface to and make use of future scalable file systems, in particular addressing quality of service guarantees. Specifically, in many scientific and engineering applications, there is a collection of sinks; each with differing needs for the same data provided by some source (e.g., the file system). Often, different sinks need different portions of the data, or they need slightly transformed data versions.

Traditionally the work to adjust data has either been done on the server (e.g., storing multiple versions of the same file) or at the sinks. The result is an increased level of data replication, and/or increased loads on recipients' CPU (central processing unit) resources, and/or increased demands on the aggregate network resources required for data transport. Our approach is to create richer interfaces between applications and data sources like the file system. The purpose is to: 1) enrich data sources with application-specific functionality that manages the data being streamed out on behalf of individual clients, and 2) take advantage of nodes "in between" the source and the sinks to perform processing and data movement actions that benefit sinks and reduce bottlenecks from resource contention.

Essentially, by automatically decomposing the transformations and generating code according to the CPU resources and network bandwidth available along the network overlay connecting sources and sinks, including the sources, sinks, and even intermediate nodes, we can combine identical operations and reduce composite CPU loads yielding overall improvements in the quality of service experienced by applications. It also affords us the opportunity to investigate other operations, such as siphoning off portions of the written or read data for immediate analysis or additional, off-line processing.

FY 2006 Accomplishments

- Studied various existing file systems and data transformation services to develop a better understanding of existing work in both of these areas.
- Developed a prototype integration of the data transformation engine with the lightweight file systems tools.
- Studied the data storage usage and file formats of typical high-performance computing applications to gain an understanding of current requirements and potential future needs for data transformation services.
- Deployed the lightweight file systems tools from Sandia into a Georgia Tech computing cluster and began educating others within the Georgia Tech community on the philosophy and use of the tools for use in their own storage-related research.
- Learned about additional high-performance computing applications that may be accessed for testing new techniques and applications of this technology.

Significance

More in-depth knowledge of various existing high-performance file systems, the Sandia lightweight file systems tools, and various high-performance computing applications affords the opportunity to focus research efforts to better suit the needs of Sandia applications. The prototype system, once complete, will ultimately lead to applications enabling more efficient utilization of resources. The techniques, in general, can lead to applications becoming more streamlined and focused on the task at hand and much less concerned with coding of data manipulation tasks. Some offline data processing can be done automatically as part of the storage operation with only a description of the required transformation, accelerating the time from an application run completing and the data being in a usable form.

Analysis of Bead Attached Ion Channels on Optically Addressable Microfluidic Electrode Arrays

86358
S. M. Brozik, G. D. Bachand

Project Purpose

In order to better understand the fundamental structural dynamics of membrane-bound ion channels and membrane-bound proteins in general, we are collaborating with the University of New Mexico to develop new solid-supported lipid bilayer platforms in which single membrane-bound proteins can be electrochemically and optically interrogated. It has recently been shown that membrane-associated proteins can be incorporated into lipid bilayers supported on silica beads. Expansion of these capabilities through high-throughput parallel and multiplex strategies offers great possibilities for biosensor technology; however, functional reconstitution of complex transmembrane proteins into this platform has yet to be demonstrated.

The introduction of functional transmembrane proteins into supported bilayer-based biomimetic systems presents a significant challenge for biophysics. Among the various methods for producing supported bilayers, liposomal fusion offers a versatile method for the introduction of membrane proteins into supported bilayers on a variety of substrates. We investigate the properties of protein contained in unilamellar phosphocholine lipid bilayers on nanoporous silica microspheres to determine the effects of the silica substrate pore structure and the curvature on the stability of the membrane and the functionality of the membrane protein. Supported bilayers on porous silica microspheres show a significant increase in surface area on surfaces with structures in excess of 10 nm, as well as an overall decrease in stability resulting from increasing pore size and curvature.

Comparison of liposomal and detergent-mediated introduction of purified bacteriorhodopsin (bR) and the human type 3 serotonin receptor (5HT3R) are investigated, focusing on the resulting protein function, diffusion, orientation, and incorporation efficiency. In both cases, functional protein with near-native diffusion constants are observed; however, the reconstitution efficiency and orientation selectivity are significantly enhanced through detergent-mediated protein reconstitution. The results of these experiments provide a basis for bulk ionic and fluorescent dye-based compartmentalization assays, as well as single-molecule optical and single-channel electrochemical interrogation of transmembrane proteins in a biomimetic platform.

FY 2006 Accomplishments

Our FY 2006 milestones were to characterize the biophysical properties of the membrane-bound proteins, bacteriorhodopsin and the serotonin type 3 receptor, on the nanoporous silica bead platform. We completed studies on the long-term stability, detergent solubilization, and protein incorporation in nanoporous microsphere supported bilayers (NMsbs) resulting from varying particle diameter and surface porosity.

In addition to traditional proteoliposome deposition, we tested a new method for introducing detergent-solubilized membrane proteins into preformed supported bilayers. We completed a comparison of the two methods based upon the incorporation efficiency, orientation specificity, and functionality. Finally, micromanipulation and patch-clamp electrochemical measurements were attempted on the NMsbs.

We made several noteworthy observations while probing the effects of substrate pore size and diameter in phosphocholine-supported bilayers. With respect to curvature, the larger diameter beads displayed the greatest capacity to maintain compartmentalized fluorescent dye and calcium ions and were the most resistant to detergent solubilization. The conclusion that high curvature in phosphocholine-supported bilayer systems can significantly reduce the resulting bilayer stability suggests the potential significance of the intrinsic curvature of the incorporated bilayer components. Thus, adjusting the lipid component mixture to more efficiently match the curvature of a specified supported bilayer system may offer a large increase in bilayer stability.

When considering pore size, we observed that in bilayers with pores less than or equal to 2x the bilayer thickness, fluid bilayers span surface structures much like a bilayer in its gel phase. When the pore sizes are much larger than the bilayer thickness, the membrane is significantly invaginated into the pore, thus increasing the membrane surface area. For various applications, different degrees of invagination may be appropriate; however, the fraction of membrane-bound protein exposed to the surface may be significantly increased and the overall bilayer stability is diminished.

In probing detergent solubilization, exposure of porous bead-supported bilayers to increasing detergent concentrations showed reversible detergent saturation of the bilayer; however, even at detergent to phospholipid ratios exceeding 50:1 (above the critical micelle concentration), we did not observe complete bilayer solubilization. To rule out the possibility of perturbations resulting from surface accessible fluorescent tags, the experiment was repeated with an in-membrane fluorescent label with the same result.

The evidence suggests, however, that the platform described is not a case of a detergent-resistant bilayer as seen in many biological systems, but possibly the result of the high surface area for mixed micelle adsorption. Finally, protein reconstitution into preformed supported bilayers was carried out at detergent saturating conditions and resulted in a large increase in reconstitution efficiency and orientation selectivity.

Significance

Porous silica bead-supported bilayers show promise for the future study of a wide range of membrane-bound proteins, providing a stable and size-selective medium that is easily interfaced with current optical and electrochemical technologies, and show promise for new chip-based sensor technology.

Single-molecule spectroscopic measurements, in combination with electrical detection, offer a unique opportunity for obtaining a dynamic view of structural/functional relationships on transmembrane proteins. However, the potential impact of combined optical and electrochemical measurements goes well beyond the study of basic biophysical mechanisms and can provide the ultimate in biodetection schemes: using the specificity of biochemical interactions, the sensitivity of single-molecule fluorescence detection, and the enormous electrochemical amplification afforded by the opening of a membrane channel.

Capture and Utilization of Prosody in Disambiguating Spoken Speech

86801
T. L. Bauer

Project Purpose

A large amount of work has gone into statistical and semantic analysis of text to support many different national security applications. However, the words themselves are only one aspect of text that people use to convey meaning in spoken dialog. This project with the University of Illinois at Urbana-Champaign investigates some of the other aspects.

The goal of the project is to gain a better understanding of how people use other types of information to deal with ambiguous speech. A critical avenue of research in this area is how people use prosody, including cues like fundamental frequency, harmonics, and amplitude. By conducting experiments investigating human use of prosodic cues, technologies may be developed that help resolve ambiguities the same way people do. Our research focuses on modeling how people use prosody to resolve ambiguity, using electroencephalography to track brain responses to sentences containing ambiguities.

FY 2006 Accomplishments

We are pursuing two related lines of research in our laboratory. The first explores the consequences of ambiguous information in language processing and cues that readers or listeners can use to navigate ambiguity. Although ambiguity is highly common in language, our studies show that readers are often unaware of it. This leads to processing difficulty and misunderstandings in which the readers never realize that they have failed to reach the correct interpretation of a statement. The potential consequences of these errors are important, so we are laying the groundwork for studies of cues, such as prosody, that could help listeners avoid ambiguity altogether.

Our second line of research also relates to errors of which people are unaware. We conducted a series of memory studies that investigate the impact of context and study strategies on memory for verbal materials. We found that readers make high numbers of memory errors when tested on lures that are similar in either meaning or visual form to words on a study list. In both cases, the readers are highly confident in saying that they had studied words which never actually appeared. However, we found that changing the context in which the words were studied had a dramatic impact on the types of lures that readers were susceptible to, probably because of differences in their study strategies when faced with different kinds of materials.

The information we are gathering from both of these lines of research is potentially useful for helping people to become aware of these unconscious errors. Once they are aware of these pitfalls, they could change their information processing strategies in order to avoid them.

Significance

These accomplishments are helping advance the basic science of understanding how people use language, specifically how they disambiguate language. As this work matures, we anticipate that it will yield an understanding of language that will help advance Sandia-developed capabilities for building models of individuals based on their speech. Increasing our ability to automatically process this type of information will help advance analysis and decision support tools and benefit national security-related programs at Sandia.

Refereed Communications

L.E. Matzen and A.S. Benjamin, “Remembering Words Not Presented in Sentences: How Study Context Changes Patterns of False Memories,” submitted to the *Journal of Experimental Psychology: Learning, Memory, and Cognition*.

Fundamentals of Embossing Nanoimprint Lithography in Polymer Substrates

93361
B. A. Simmons

Project Purpose

Nanoimprint lithography (NIL) is a nanomanufacturing technique that uses a nanostructured master template to form features in a substrate with best resolution near 1 nm and areas as large as 100 cm^2. In embossing-based NIL, a nanostructured master is heated and pressed into a thermoplastic polymer film, forming a negative relief replica in the polymer film. Nearly all previous research on NIL has been for the purpose of nanoelectronics fabrication in which the polymer film acts as a mask layer. We seek to develop the embossed, nanostructured polymer film as the functional surface, rather than a mask layer.

The goal of the project is to develop a first principles understanding of polymer transport during printing such that any polymeric material could be employed for embossing-based NIL. This project is a joint collaboration with PECASE recipient William King of the Georgia Institute of Technology. The need for this research is particularly acute when polymer nanostructures are of size comparable to the polymer molecule radius of gyration, and when the nanoscale heat and mass transport properties of the polymer are not known.

The lack of fundamental materials science knowledge is the major limit to rational design of nanofabrication methods designed to produce specific feature sizes and shapes. This work aims to model and measure polymer properties during NIL for a large number of thermoplastic polymers. This link between material properties and processing parameters will enable rational NIL process design.

FY 2006 Accomplishments

This activity is investigating nanoscale polymer heat and mass transport that occurs during embossing-based NIL and direct-write atomic force microscopy (AFM) nanoindentation. Master templates with features in the range 2 micron – 10 nm have been manufactured in silicon and electroplated into Ni metal. These Ni stamps were formed to accommodate many embossing trials with minimal deformation, as compared to other soft-lithography materials. A number of thermoplastic polymers have been investigated, with molecular weights in the range 10^4-10^7 kDa, corresponding to characteristic molecular lengthscales in the range 3 – 50 nm. These results served as the initial training set to optimize design rules for advanced stamp designs.

AFM has been used to characterize the embossed substrates and correlate embossing conditions, polymer molecular properties, and manufactured feature sizes. Continuum and subcontinuum models of polymer transport and template deformation have proven beneficial in this analysis. The ultimate goal of this project is to establish design rules for the manufacture of polymer nano/microdevices, and we succeeded in the initial stages of this project in developing the toolbox necessary to realize such designs in the next stage of this project.

In addition to developing these design rules for NIL, we also investigated the interaction of patterned surfaces and cellular adhesion. The major goal of this aspect of the research is to determine the impact of topographical length-scales on the interaction between a cell and a surface. The results obtained from this research were used to develop next-generation tissue scaffolds and for mitigation of biofouling, and were demonstrated by attaching osteoblasts in predetermined rational patterns.

Significance

NIL is the ultraminiaturized version of the decades-old embossing process in which a master tool or mold is pressed into a soft material to create detailed patterns. Using a broad range of polymer materials, NIL produces structures on the micron or nanometer size scales, offering the potential for lowering production costs. However, quality issues caused by unpredictable polymer flow into the nonuniform features of embossing tools pose a major stumbling block. Earlier research into this complex process produced often conflicting recommendations, forcing manufacturers to pursue costly trial and error methods.

Using the results of experimental work, we examined every variable involved in the nanoimprinting process and recorded the outcome of each incremental change through the design space. We studied shear deformation of the polymer, elastic stress release, capillary flow, and viscous flow during the filling of imprinting tool cavities that had varying sizes and shapes. The results apply to any polymeric material that follows standard viscous flow rules and produces feature sizes larger than 50 nanometers. The next step in this research will be to modify the simulation software to account for physics changes that occur on smaller size scales.

These results will have applications in semiconductor manufacturing, where nanoimprinting offers a potential alternative to increasingly expensive lithography processes to produce circuitry. It could also help make high-volume production of nanoscale structures for optoelectronic, biomedical, and other applications more economically feasible.

Refereed Communications

J. Charest, M. Eliason, W. King, A. Talin, and B. Simmons, “Polymer Cell Culture Substrates with Combined Nanotopographical Patterns and Micropatterned Chemical Domains,” *Journal of Vacuum Science and Technology B*, vol. 23(6), pp. 3011-3014, November 2005.

Rational Understanding and Control of the Magnetic Behavior of Nanoparticles

93362

B. A. Simmons

Project Purpose

Magnetic spinel ferrite nanoparticles, MFe_2O_4 (M = Mn, Mg, Zn, Co, Fe, and so on), have great potential for biomedical diagnostics and targeted drug delivery. To realize biomedical applications of nanoparticles, the magnetic properties of the nanoparticles must be optimized and the surface of the nanoparticles must be modified with biocompatible ligands and/or polymer matrices that can also carry medicinal drugs. These two requirements are interconnected.

Nanoparticle coercivity is strongly dependent on the surface pinning of magnetic moments caused both by metal cation coupling and by magnetic surface anisotropy. Different surface metal cations possess different intrinsic coupling strength and will result in markedly different coercivities. For example, Co ion usually possesses a strong quantum coupling between electron spin and atomic orbital angular momentum. However, Mn ion has a weak coupling. Thus, Co terminated nanoparticles should display larger coercivity when compared to Mn terminated nanoparticles.

Surface modification of nanoparticles also profoundly alters the coercivity of nanoparticles. We hypothesize that the surface modification induced changes in coercivity are caused by missing coordinating oxygen atoms around surface metal cations. When the coordination of surface metal cations is similar to the coordination symmetry of the metal cations in the core of a nanocrystal, the surface magnetic anisotropy, the resulting surface pinning, and the coercivity should be low. Thus, the observed dependence of the coercivity on surface modification is likely due to changes on the coordination of surface metal cations caused by the chemical surface modification.

We will study the correlations between the coercivity of nanoparticles, the quantum couplings in surface metal atoms, and the surface modification. The research will be conducted at the Georgia Institute of Technology with PECASE recipient Z. John Zhang.

FY 2006 Accomplishments

We systematically investigated the superparamagnetic properties of $CoFe_2O_4$ and Fe_3O_4 nanocrystals. The observed blocking temperature of $CoFe_2O_4$ nanocrystals is at least 100 degrees higher than that of the same sized Fe_3O_4 nanocrystals. The coercivity of $CoFe_2O_4$ nanocrystals at 5 K is over 50 times higher than the same sized Fe_3O_4 nanocrystals.

We observed that the drastic difference in superparamagnetic properties between the similar sized spherical $CoFe_2O_4$ and Fe_3O_4 (or $FeFe_2O_4$) spinel ferrite nanocrystals was correlated to the coupling strength between electron spin and orbital angular momentum (L-S) in magnetic cations. Compared to Fe^{2+} ion, the effect of much stronger spin-orbital coupling at Co^{2+} lattice sites leads to a higher magnetic anisotropy and results in the dramatic discrepancy of superparamagnetic properties between $CoFe_2O_4$ and Fe_3O_4 nanocrystals.

Significance

This work provides insight to the fundamental understanding of the quantum origin of superparamagnetic properties. Furthermore, these results suggest that it is possible to control the superparamagnetic properties through magnetic coupling at atomic level in spinel ferrite nanocrystals for various applications. The ability to control magnetic properties is vital for the development of new magnetic materials for storage and sensing.

Refereed Communications

Q. Song and Z.J. Zhang, "Correlation Between Spin-Orbital Coupling and the Superparamagnetic Properties in Magnetite and Cobalt Ferrite Spinel Nanocrystals," *Journal of Phys. Chem. B*, vol. 110, pp. 11205-11209, June 2006.

E.L.H. Heintz, P. Rohatgi, D.F. Doyle, C.J. Fahrni, and Z.J. Zhang, "Magnetic $CoFe_2O_4$ Nanoparticle-Oligonucleotide Conjugates for Cellular Uptake and Magnetic Manipulation," to be published in the *Journal of American Chemical Society*.

MEMS Dual-Backplate Capacitive Microphone

93364
W. K. Schubert

Project Purpose

The purpose of this project is to develop a dual-backplate microphone using Sandia's ultraplanar, multilevel MEMS Technology (SUMMiT V™) fabrication process. A dual-backplate microphone consists of a diaphragm and two porous backplates, one on either side of the diaphragm. These three plates are conducting, thus creating two capacitors. The holes in the backplates allow the incident pressure to pass through the backplates and impinge on the diaphragm.

As the incident pressure deflects the diaphragm, the capacitance of the top and bottom capacitors change. An electrical bias is applied to the microphone; therefore, the change in capacitance causes an electrical output. This can be either a voltage or a charge depending on how the microphone is biased. The microphone is packaged with interface circuitry to amplify the output and produce the buffered output voltage.

The fabrication of this device presents several challenges. First, three independent conducting layers must be fabricated to construct the diaphragm and the two backplates. The diaphragm should be compliant to increase the sensitivity of the device. The thickness of the gap between the plates, as well as the thickness of the plates themselves, must be uniform and well-controlled.

The SUMMiT V fabrication process is unique in that it can meet the fabrication needs of this project. The SUMMiT V process has been optimized to provide compliant mechanical layers that are ideal for the construction of the microphone's diaphragm. Furthermore, the use of chemical mechanical polishing results in extremely flat structural layers and uniform spacing between the layers, both of which are critical to the successful fabrication of the MEMS microphone.

This microphone is designed for aeroacoustic applications, which have vastly different specifications than typical audio microphones. An aeroacoustic microphone should be capable of operating up to sound pressure levels (SPL) of 160 dB due to the high SPLs radiated by jet engines. Furthermore, the frequency range of interest for aeroacoustic measurements extends up to 90 kHz because experiments are often conducted on scale models. In order to have diffraction-free measurements at high frequencies, the microphone size must be small; at 90 kHz, the microphone radius should be less than 0.6 mm. MEMS microphones are well-suited for this application because of the desired small size and high frequency range.

The majority of previous research in MEMS microphones has focused on audio microphones. However, this project will improve on previous MEMS-based aeroacoustic microphones and is the first dual-backplate capacitive MEMS microphone developed for aeroacoustic applications.

FY 2006 Accomplishments

We focused on documentation of research results and the release of more microphones. We developed a complete electromechanical lumped-element model for the dual-backplate microphone. In addition, we developed a noise model for the microphone and completed the analysis for the microphone packaged with a charge amplifier. To enable the reliable packaging of the released microphones, we modified the release process to include bond pad metallization. Initial attempts to wire bond to the polysilicon bond pads proved unreliable and resulted in many damaged devices. The bond pad metallization is not trivial, as the metal is exposed to the release etchant. This etchant is some form of HF mixture to etch the sacrificial oxide; initially, 49 percent HF was used for the release etch.

The first metallization we attempted was gold with a chromium adhesion layer. These metals were chosen because of their resistance to HF. However, devices released with the Au/Cr metallization did not function properly. After a thorough investigation of the device failure, including a literature search and analysis using scanning electron microscopy, we determined that the presence of gold increases the HF etch rate of polysilicon. Based on the literature, we believe that Au nanoparticles released by the Au metallization act potentially as a catalyst for HF. This results in damage to a critical feature of the microphone, an electrical connection from the bottom backplate to the bond pad in POLY0.

The latest work focused on developing a release process that utilizes aluminum bond pads. However, typical HF solutions readily attack aluminum. We found that an oxide etchant, Silox Vapox III produced by Transcene Company, Inc., provides a sufficient etch rate for oxide while providing a selectivity of about 50:1 to aluminum. Initial testing shows that this etchant should be able to successfully release the microphone with aluminum metallization.

Significance

There are several contributions of this research to the general scientific and technical community. As part of this work, we completed detailed modeling of a dual-backplate microphone. A lumped element model for a dual-backplate microphone was developed. This model considers the effects of the microphone structure and includes the transduction elements to give an electrical output. We also developed a noise model of the microphone. This can be analyzed with either a voltage amplifier or charge amplifier to study the noise sources of a dual-backplate microphone. These models will be useful to researchers developing future dual-backplate microphones for any of a variety of applications, including those specific to national security issues.

This device shows the feasibility of a dual-backplate capacitive microphone for aeroacoustic measurements. The designed microphone has achieved a high bandwidth and a wide dynamic range. Furthermore, this device demonstrated a novel use of the SUMMiT V process and the viability of this process for MEMS microphones.

Refereed

D.T. Martin, J. Liu, K. Kadirvel, R.M. Fox, M. Sheplak, and T. Nishida, "Development of a MEMS Dual-Backplate Capacitive Microphone for Aeroacoustic Measurements," presented at the 44th AIAA Aerospace Sciences Meeting and Exhibit, Reno, NV, January 2006.

J. Liu, D.T. Martin, K. Kadirvel, T. Nishida, L. Cattafesta, M. Sheplak, and B.P. Mann, "Nonlinear Model and System Identification of a Capacitive Dual-Backplate MEMS Microphone," to be published in the *Journal of Sound Vibration*.

Other Communications

J. Liu, D.T. Martin, K. Kadirvel, T. Nishida, M. Sheplak, and B.P. Mann, "Nonlinear Identification of a Capacitive Dual-Backplate MEMS Microphone," presented at the 2005 ASME International Design Engineering Technical Conferences, Long Beach, CA, September 2005.

J. Liu, D.T. Martin, K. Kadirvel, T. Nishida, L.N. Cattafesta, M. Sheplak, and B. Mann, "Nonlinear System Identification of a MEMS Dual-Backplate Capacitive Microphone by Harmonic Balance Method," presented at the 2005 ASME International Mechanical Engineering Congress and Exposition, Orlando, FL, November 2005.

Process Science and Engineering for Thermomechanical Nanomanufacturing

93366
A. C. Sun

Project Purpose

Embossing and molding are simple techniques for molecular-scale replication. Recent studies showed replication of single-walled carbon nanotubes with diameter 2 nm via molding and crack tips of size 0.4 nm via casting. Nanoimprint lithography (NIL) offers scalable embossing or molding of sub-10 nm features over large areas.

In the nanoimprint embossing process, a nano-patterned mold is heated and pressed into a thermo-plastic polymer film, replicating a negative relief of the mold in the polymer sample. Often, the replicated features are close in size to the polymer radius of gyration (R_g). Atomic force microscope (AFM) nano-indentation is one type of nanoembossing process whereby a sharp conical tip forms indents into a thin polymer layer. As film thickness and feature sizes shrink to 20 nm in AFM nanoindentation, polymer thermomechanical behavior may deviate from that of bulk.

Reliable design of molecular-scale molding processes will require a deep understanding of fundamental polymer mechanics occurring at the nanometer level. This work presents continuum GOMA simulations that model polymer flow during AFM nanoindentation where an atomically sharp tip of specified radius of curvature indents thin films of high molecular weight (MW) polymer. The simulations identify bulk polymer thermomechanical properties that govern nanometer-scale polymer transport in isothermal and nonisothermal AFM nanoindentation. This work is being performed in collaboration with the George Institute of Technology.

FY 2006 Accomplishments

Simulations predict isothermal polymer flow in 35 nm 350k MW polymethylmethacrylate (PMMA) film atop a silicon substrate due to constant velocity indentation of a silicon AFM tip of radius 20 nm. Simulations of AFM nanoindentation into the Carreau Williams-Landel-Ferry polymer produce both local and nonlocal deformation, dependent on shear rate and temperature.

For indents at a given temperature, deeper penetration depths result in higher polymer shear rates. Polymer shear rates resulting from constant velocity indentation remain nearly constant as temperature changes, but the localized viscosities move from the Rouse viscosity transition region at low T to the power law rubbery regime at higher T. Force-distance curves from simulation well match reported experimental force-distance curves of polydisperse PMMA over a range of temperatures.

The modified continuum simulations accurately predict subcontinuum polymer mechanical deformation due to an AFM tip of radius ~ R_g indenting a polymer film from initial thickness above the polymer molecular diameter 2 R_g to below the unperturbed R_g. The agreement of simulation force-distance curves and indentation slope to measurements reported in literature suggests that material deformation for high MW polymer films during isothermal AFM nanoindentation closely follows shear-thinning bulk material behavior with a full-slip polymer-tip interface. The continuum simulations allow for optimization of AFM nanoindentation processes based on bulk viscoelastic material properties and extend continuum descriptions of polymer mechanics to length scales below R_g.

The continuum material model developed for isothermal AFM nanoindentation also investigated polymer deformation and heat transfer during nonisothermal AFM thermomechanical data bit formation. The simulations show highly localized deformation in the temperature dependent shear-thinning polymer. The tip locally heats the polymer, creating a temperature gradient spanning from the specified tip temperature to room temperature over radial distance ~ 50 nm from the polymer-tip interface. The resulting temperature dependent complex viscosity ranges orders of magnitude over the same radial distance.

The steep spatial gradient of the temperature shift factor results in highly localized polymer deformation resembling lubrication, with only the most mobile polymer layer at highest temperatures deforming. Applied load only slightly affects the rate of indentation as the temperature-dependent viscosity dominates the nanoindentation process. The simulations well match measurements of nonisothermal AFM indentation into 600k MW PMMA at cantilever heater temperature 380 °C. The simulations suggest Tint ~ 240 °C predicts measured material response and 25 °C increments in Tint significantly impact timescale of indentation.

Simulations of nonisothermal AFM nanoindentation with material properties based on bulk polymer physics model experimental measurements and explain high-cantilever heater temperatures required for indentation formation due to localized polymer deformation. The simulations present a modeling tool to further compare with experiment to inform subcontinuum heat transfer models of polymer-tip interface temperature and characterize the bit writing process.

Significance

This work is synergetic with several mission-critical areas within Sandia's science and technology community. First are the advances in nanotechnology research and second, the advances in simulation technology. Nanoembossing using an AFM tip is already revolutionizing the field of lithography. The ability to understand the material response based on continuum-based simulations will enable us to engineer materials that would have required experiments that are expensive to perform.

Refereed Communications

H.D. Rowland, W.P. King, A.C. Sun, and P.R. Schunk, "Simulations for Process Design of Nanoembossing and Atomic Force Microscope Thermomechanical Nanoindentation," in *Proceedings of the 2005 MRS Fall Meeting*, p. 200, November 2005.

Fabrication and Device Applications of Aligned Mesoporous Architectures

93369
C. J. Brinker

Project Purpose

A well-known problem in self-assembled silica/surfactant thin film mesophases and corresponding calcined mesoporous silica films is that two-dimensional hexagonal mesophases are oriented parallel to the substrate surface. Monosized pores oriented normal to the substrate surface are important for control of molecular and ion transport in synthetic membranes and for control of charge and energy transfer in photovoltaics and organic light-emitting diodes (OLEDs).

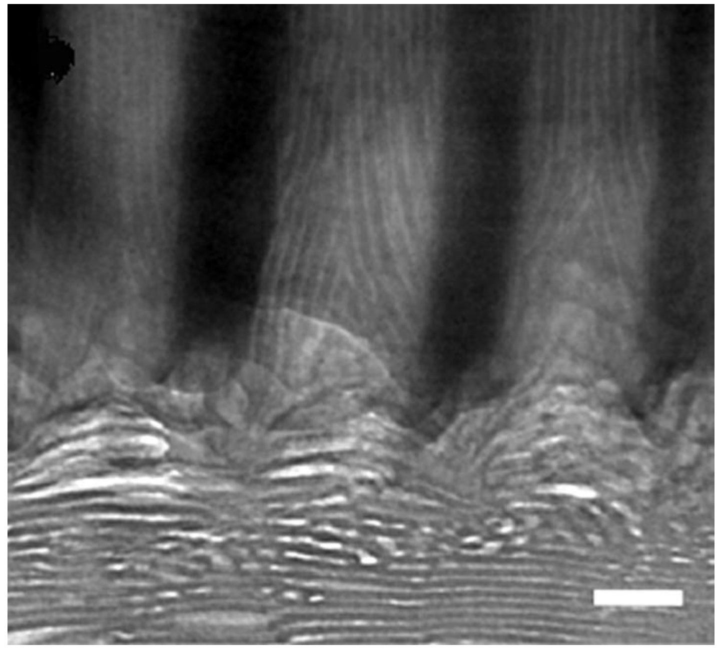
To date normally oriented channels have been formed by suction of sols through anodized alumina membranes, but drying and associated shrinkage lead to defects and irreproducibility. To overcome these issues, we will explore the capillary filling of preformed silica/block copolymer mesophases into alumina nanochannels.

The purpose of the project, a collaboration with PECASE recipient Yunfeng Lu at Tulane University, is to understand the mesophase transformation and/or reorientation under capillary force and to develop oriented mesochannels that are normal to the substrate surface. These materials are interesting for the applications of separation, highly sensitive sensing, and smart devices.

FY 2006 Accomplishments

In FY 2005, we demonstrated that self-assembled hexagonal mesophases can be aligned by capillary forces. By contacting a liquid crystalline hexagonal silicate/copolymer composite film with porous anodized alumina membranes, the liquid crystalline silicate/surfactant mesophases are gradually filled into the cylindrical alumina pores driven by the capillary force, resulting in oriented hexagonal mesophases normal to the substrate surface.

In FY 2006, we successfully captured the mesophase transformation at the interface between the preformed film and the alumina membrane by cross-sectional transmission electron microscopy (TEM). The parallel to normal reorientation of the cylindrical channels is well demonstrated by the cross-sectional TEM image.



Cross-sectional TEM image of oriented mesoporous silica nanowire/alumina membrane shows mesostructure transition at the interface between pre-formed mesoporous silica film and the alumina substrate.

Over a region 50-100 nm thick (depending on diameters of alumina nanochannels), the preformed hexagonal mesostructure is gradually reoriented by shearing force to a direction normal to the substrate. We systemically studied the effect of diameter variation of alumina nanochannels on the mesostructure orientation and identified the critical diameter for producing the oriented mesoporous silica nanowires.

Hexagonal mesophases are predominantly aligned along the silica nanowires using the alumina membranes with different channel diameters ranging from 200-30 nm. By tuning the diameter of the alumina nanochannels, the number of aligned mesopores within an oriented silica nanowire is therefore controllable. For example, a 30 nm silica nanowire only contains 2-3 straight oriented mesopores. When the diameter of the alumina nanochannels is reduced to 20-30 nm, the silica mesophase is no longer fully aligned. In the region where the diameter is 20 nm, a curved tubular mesophase is formed. However, in the region where the diameter is 30 nm, a short aligned tubular mesophase is formed indicating that 30 nm is the critical diameter of alumina nanochannels to produce oriented mesostructures by capillary filling.

Significance

Success of this work will provide new platforms for extensive use in water purification, separation, sensors, templated synthesis, microelectronics, optics, controlled release, and highly selective catalysts. For example, aligned mesoporous materials can be further functionalized with responsive or recognizable components, leading to the fabrication of smart devices, such as intelligent artificial membranes and highly sensitive sensors.

This work supports the DOE science strategic goal of maintaining a world-class research capability; e.g., advances in nanoscale science built around foundations in materials and chemistry that may lead to improved energy technologies and systems.

Other Communications

R. Kou, D. Wang, and Y. Lu, "Oriented Composite Mesostructures under Nanoscale Confinement," presented at the AIChE Annual Meeting, San Francisco, CA, November 2006.

Atmospheric Aerosols

93652
B. D. Zak

Project Purpose

The purpose of this project is to develop a new "smog chamber" technique for investigating the formation and evolution of ambient aerosols from primary gaseous air pollutants, and/or the evolution of a known primary aerosol when exposed to gaseous air pollutants from the ambient environment. This project is in collaboration with Crystal Reed, graduate student at Texas A&M University.

Atmospheric aerosols have major impacts on climate, both directly through scattering and absorption and indirectly through cloud processes. Understanding these impacts can be challenging given the complex nature and composition of the atmospheric aerosol. In order to better understand the effects of aerosols on the environment, we must study the processes of aerosol transformation in the ambient environment. Such processes include the transformation of soot, which can result in changes occurring to the radiative properties of black carbon and alter our understanding of its effects on the environment.

Another process of concern includes the formation of secondary organic aerosols in which an otherwise hygroscopic particle may undergo changes via the condensation of nonhygroscopic organic vapors. During this transformation, the hygroscopicity of the particle may decrease, leading to an alteration in cloud microphysical processes.

Aerosol chambers have been used in previous laboratory experiments to investigate the coating of gas phase species on existing aerosols. However, laboratory results may not be completely representative of the ambient environment. Captured air experiments, which are more representative of the ambient environment, involve trapping ambient air in several identical chambers and injecting a single compound with varying concentrations into successive chambers in order to investigate the impacts of the compound addition.

However, due to the closed nature of the system, processes that take an exceptional amount of time will not be accurately represented since the captured air concentration will decay with time. Therefore, the purpose of this project is to develop a new chamber type, the ambient aerosol chamber for evolution studies (AACES), in order to better understand the ambient effects on aerosol transformation. AACES construction will allow for measurement periods beyond 24 hours without depleting the gas concentration within the chamber.

FY 2006 Accomplishments

We completed a prototype of the AACES in early April, 2006. AACES is a roughly cubical chamber constructed of a rigid Acrylite OP-4 acrylic outer shell, which transmits ultraviolet (UV) radiation both in the UV-B (280-215 nm) and UV-A (315-400 nm) ranges. FEP Teflon lines the inside of the chamber on all sides and the top, while expanded PTFE (ePTFE) Teflon is used on the bottom of the chamber. The fibrous structure of the ePTFE acts as a barrier to particulates, while allowing gas molecules to move virtually unimpeded from one side of the chamber wall to the other, creating an initial environment inside the chamber that is free of particles and continuously mimics the ambient air.

In order to create an environment within the chamber that is well-mixed, we discovered a new environmental chamber mixing technology. Internal Teflon coated fans have been the preferred method of mixing in environmental chambers. However, due to their invasive nature, such fans lead to an increase in surface area within the chamber which may lead to an increase in depositional loss. Through the use of subsonic sound at 20 Hz using an external, noninvasive 12-inch subwoofer, we were able to completely mix the chamber efficiently with little effect on the particle loss rate.

We conducted a series of tests to verify AACES ability to perform in the field:

Particle Filtration
PTFE membrane has an estimated 96 percent filtration efficiency for particles larger than 10 nm under typical filtration conditions. The particle filtration efficiency as tested under chamber conditions resulted in over a 99 percent removal of ambient particles. This was tested using a TSI 3760A Condensation Nucleus Counter.

Ambient Gas Penetration Efficiency
We tested ozone penetration efficiency using a Thermo Electron Corp. O_3 Gas Analyzer. Since ozone is highly reactive, its penetration efficiency across the ePTFE membrane reveals the general penetration efficiency of many other species. Resulting ozone concentration within the chamber was above 90 percent of ambient.

Particle Deposition/Wall Loss
Many processes that lead to particle evolution have time scales on the order of hours to days. Therefore, we tested the depositional loss of particles within the chamber. First, the chamber was completely flushed with zero air to ensure an initial clean environment. Second, we injected 70 nm uncharged ammonium sulfate particles, and monitored the concentration on an hourly basis using a differential mobility analyzer (DMA). In order to inject only uncharged particles, a tandem DMA was used. Ammonium sulfate particles generated using a TSI atomizer were first dried, passed through a charger, and size-selected using the first DMA. The size-selected particles were then passed through a charger prior to entering the second DMA set at a high voltage sufficient to remove all charged particles. Particle loss rate was less than 10 percent per hour.

Upon the successful completion of all chamber tests, a second chamber was created and AACES deployed in the field in August 2006.

Significance

Numerous studies have used laboratory and smog chamber results in model analysis to predict the behavior of the atmospheric aerosol. However, the simplistic treatment of aerosols in models, coupled with the difficulty in interpretation of ambient aerosol measurements due to source variability, makes a comparison between model results and measurements difficult at best. By introducing a monodisperse aerosol of known composition into the AACES, we can monitor the growth rate due to such processes using a DMA system. With this new knowledge, chemistry models will be better equipped to predict the behavior of the atmospheric aerosol. Furthermore, results from these studies will aid the development of future aerosol modeling techniques designed for comparing the ambient aerosol with model predictions.

With the growing concern regarding organic aerosol concentrations, the effects of low volatile organic species on aerosol properties can be better understood by monitoring changes occurring to the hygroscopic aerosols over time. Through the use of AACES, we will be able to separate the hygroscopic growth of an aerosol from variability in source region and history prior to sampling, providing a better understanding of the ambient effects on aerosols, including soot, and increase the certainty of aerosol classification and composition inferred from hygroscopic growth measurements. Also, field studies often result in data that is difficult to interpret due to the variability in both the temporal and spatial scales. Through collaborative efforts, AACES may be utilized in the interpretation of such field data.

Internal mixing devices implemented in environmental smog chambers create both design challenges and the opportunity for leaks. Furthermore, the placement of these devices may create concerns with turbulent mixing and particle loss rates. By implementing the newly developed subsonic mixing technology, future Teflon chambers will be more efficiently constructed and better equipped to more accurately represent heterogeneous reactions in the ambient environment. Moreover, the use of subsonic sound to move air will open new doors to innovation and technology.

The techniques developed here are applicable to hazardous aerosols that are of concern to Sandia: routine aerosol emissions from new or modified energy systems; routine aerosol emissions from new or modified elements of the nuclear fuel cycle; and accident-related aerosols from either the nuclear fuel cycle or nuclear weapons.

Human Interaction with Safety-Critical Interconnected Systems

94808

R. J. Glass Jr., R. A. Laviolette, M. M. Oishi

Project Purpose

The attentive driver of a car interacts with a control system that consists of a wheel, accelerator, brakes, gear shift, and so on. In spite of many advances, one can still find a way to turn a car onto its roof even without leaving a paved road. One of the active areas of research in control systems theory is the design of a control system that is guaranteed to operate within specified bounds. Even for purely automated systems, the problem is complicated, but it becomes even more so for hybrid systems, in which humans interact with automated systems.

This work is intended to develop first: general, formal one-step (in contrast to current multistep) methods to find hybrid control systems whose behavior is guaranteed bounded, and second: to develop formal methods for the recovery from error by a hybrid control system. It will turn out that both can be simultaneously achieved through a reachability analysis of hybrid control systems, which until recently was thought to be intractable for nontrivial systems.

FY 2006 Accomplishments

We obtained novel contributions in reachability analysis for human interaction with complex systems. We have presented a method to determine, through a Hamilton-Jacobi reachability computation, the set of states in safety-critical systems which will reach the desired equilibrium without saturating the input or violating the state constraints. Thus both envelope protection and stabilization under saturation are simultaneously achieved. This involves a reachability analysis on an extended state space that incorporates a parameter from the feedback linearizing input. By incorporating the input saturation, stability, and state constraints simultaneously in the initial cost function, the resultant invariant set will be the largest set of states, given bounded input, that will stabilize the system and always remain within a given constraint set.

The work contributes to the difficult problem of determining stabilizing controllers for safety-critical systems under nonlinear state and input constraints. We formulated the recovery problem for hybrid systems with flexibility in continuous inputs, discrete inputs, or state constraints. Standard reachability analysis will reveal those states from which failure is avoidable with the proper choice of control law. In the event that failure does occur, a new forward reachability calculation can identify those failure states from which recovery is possible, as well as the control input (both continuous and discrete) necessary for that recovery.

This new calculation exploits the flexibility inherent in the hybrid system – if this flexibility were not present, a recovery calculation would provide no new information from the initial backwards reachability calculation. The recovery calculation involves temporarily adjusting the system's constraints in order to recover to standard operation and standard constraints. The forward reachability calculation yields not only the forward reachable set from error states, but also the control law required to achieve that set.

Two real-world examples were presented to illustrate both methods: 1) longitudinal aircraft dynamics, and 2) two-aircraft lateral collision avoidance dynamics. The dynamics for both examples are derived from physical models of civil jet aircraft. These two case studies provide interesting motivation for further work in synthesizing recovery maneuvers. Future work will proceed in comparing the forward reachability result with a converse problem in which the standard operating region is propagated backwards in time under the new recovery dynamics.

Significance

These contributions were inspired by real-world problems in aircraft, but are likely to arise in other complex systems, including biomedical devices, driver-assistance programs, nuclear surety, the power grid, and other critical infrastructures. Many future directions of work are possible, including 1) minimization of the number of switched, nonsaturating controllers when multiple solutions to the control parameterization problem are possible; 2) alternative, less computationally exhaustive formulations to sample the parameter space; and 3) one-step synthesis of a minimal number and optimal selection of input parameters for switched, nonsaturating, feedback linearizing controllers.

Presentation and publication of some of this work in the 45th IEEE Conference on Decision and Control" won a best paper award.

Refereed Communications

M. Oishi, I. Mitchell, C. Tomlin, and P. St.-Pierre, "Computing Viable Sets and Reachable Sets to Design Feedback Linearizing Control Laws Under Saturation," in *Proceedings of the 45th IEEE Conference on Decision and Control*, February 2006.

M. Oishi, "Recovery in Flight Management Systems: Applications of Hybrid Reachability," in *Proceedings of the IEEE Advanced Process Controls Industrial Applications Workshop*, May 2006.

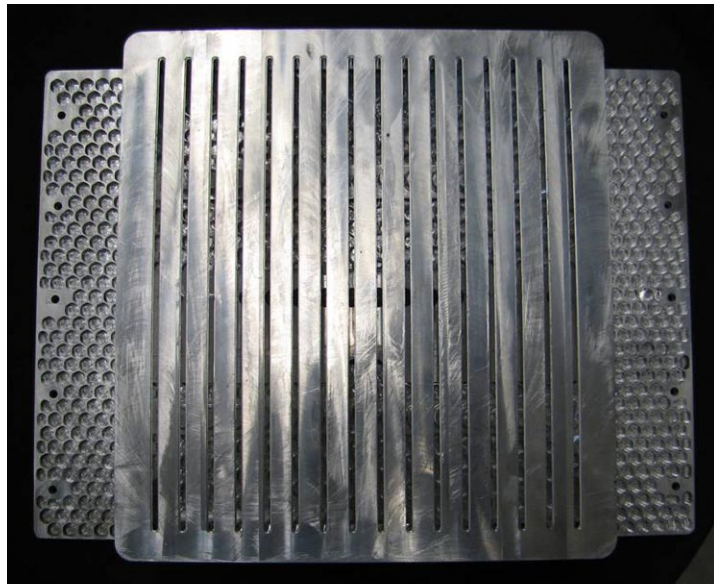
Process and Infrastructure Development for Integrated Three-Dimensional Mesomanufacturing

94809

J. A. Palmer, B. D. Chavez

Project Purpose

The purpose of this project with the University of Texas at El Paso (UTEP) was to explore ultrasonic consolidation (UC) layered manufacturing as a possible manufacturing technology for building smaller and lighter steerable antennas of interest to Sandia. UTEP provided expertise in direct write (DW) of conductive inks so that DW could be combined with UC where applicable to successfully accomplish the antenna assembly. The UTEP research was divided into two focus areas of research: tensile strength of direct write inks, and ink gap depth penetration.



Integrated antenna body built layer by layer using ultrasonic consolidation.

FY 2006 Accomplishments

- Designed tensile test specimens to determine strength of DW inks for securing electrical conductors
- Inspected tensile test specimens (pre- and post-cure and fracture)
- Performed tensile tests for two DW inks and a standard solder connection
- Determined the effect of slot width (gap) on ink penetration
- Determined the mechanism (or mechanisms) that affect penetration depth

Significance

We conducted this research in coordination with the Sandia team on the passive electronically steerable array (PESA) for miniature synthetic aperture radar (miniSAR) LDRD project 67028. PESA represents a potential novel application of a prior joint Sandia/UTEP LDRD effort in rapid prototyping of high density circuitry (RPHDC) in which a SAR assembly may be practically consolidated, miniaturized, and functionally improved by introducing low-mass stereolithography (SL) support members, and ultimately a monolithic body with encapsulated interconnect.

UTEP investigated design and manufacture of hybrid SL-DW radio frequency (RF) interconnect in this application. The interconnect was created using an integrated SL-DW apparatus we developed. This led to a separate FY 2006 LDRD project on integrated manufacturing of a MEMS antenna (project 102606), and the first known research in the area of integrated DW and novel ultrasonic consolidation, an aluminum-based solid freeform fabrication (SFF) technology.

This research is significant in that it represents an unprecedented (to the authors' knowledge) application of integrated, ambient-temperature, metallic solid freeform fabrication technologies (three-dimensional printing) in manufacturing of electro-optomechanical and RF systems. Dimensional uncertainties that compound as the square root of the sum of the squares of individual part tolerances are reduced or eliminated by the monolithic approach, thus leading to reduced dimensional compensation and therefore reduced labor cost.

The significance of this effort to the war fighter and the taxpayer is faster weapon system delivery at lower manufacturing cost through measurable reductions in parts, material, labor, and scrap. For the Department of Defense, this would also mean a decreased burden for material review board and disposition activities.

Refereed Communications

C.J. Robinson, B.E. Stucker, A.J. Lopes, R.B. Wicker, and J.A. Palmer, "Integration of Direct-Write (DW) and Ultrasonic Consolidation (UC) Technologies to Create Advanced Structures with Embedded Electrical Circuitry," in *Proceedings of the 2006 Solid Freeform Fabrication Symposium*, August 2006.

A. Lopes, M. Navarrete, F. Medina, J.A. Palmer, E. MacDonald, and R.B. Wicker, "Expanding Rapid Prototyping for Electronic Systems Integration of Arbitrary Form," in *Proceedings of the 2006 Solid Freeform Fabrication Symposium*, August 2006.

Reliable and Secure Communication in Wireless Sensor Networks

94810
N. M. Berry

Project Purpose

This research addresses the general problem of how to efficiently coordinate multiple inputs to a single-source computational unit. This general problem is more complex in sensor networks where reduced bandwidth and increased efficiency in the network are critical to the successful performance of the system. We consider the optimal compression achievable in sensor networks where these resource constraints hamper the overall design of the sensor network coordination process.

There are two main avenues of compression. First, one can exploit the correlation between the data collected at the various nodes. Second, one can exploit the relative simplicity of the function to be computed at the receiver side. Slepian and Wolf made precise the exploitation of the correlation in the data. However, contemporary distributed data-compression schemes fail to account for the receiver-side goal, often the computation of a simple function of the data received. This research will address this problem to make precise the exploitation of the function and implement such a compression scheme.

This compression scheme will probably introduce a small probability of error. We seek to better understand of this probability of error in order to ensure it will always be "small." This will involve more extensive simulations of the scheme with more varied functions and more than two sources.

Understanding the types of functions that give better compression is a primary component of this research. Certainly, a one-to-one function gives no coding gain and the scheme is not worth implementing under these conditions. But, between such a function and a unary function, there is quite a bit of variability. We will develop a means to classify functions as simple enough or not simple enough to use this scheme approach.

This work will be performed in collaboration with the Massachusetts Institute of Technology.

FY 2006 Accomplishments

We concentrated on narrowing the focus of this research and began to design and develop the methodology for how the data compression will be accomplished.

Algorithm design is based on the following steps:

1. Based on the knowledge of the given function, use graphs to distinguish amongst the function values.
2. Given a function and the statistics of a set of sources, construct a graph for each source that carries all the information relevant to distributed encoding. Each vertex for the graphs would identify with a particular source word. The compression then comes from the idea of coloring the vertices of the graph. The graph is constructed in such a way that the colors are sufficient statistics of the sources.

The two-part scheme, using two binary functions, was simulated using vehicle tracking data. The results showed coding gains of twenty percent or more over contemporary methods when using a simple greedy coloring algorithm (with probability of error less than 0.1 percent).

Significance

There are two key accomplishments for this initial year of research starting with the scoping of the problem space to deal with distributed data compression. The initial problem statement for this research broadly covered numerous communication issues from the distributed sensor networks field. By narrowing the focus, we began to address a major difficulty associated with the resource limitations of these distributed sensor networks. The initial development of methodology to explore how to share large amounts of data across limited bandwidths is the second key accomplishment of this research. The eventual results of this research will benefit researchers focusing on multiple-input-single-output systems, where data needs to be compressed in a distributed manner.

Within the Sandia mission space, this research is applicable to the many distributed systems used to address security of nuclear weapons, borders, and simulations that deal with vast data repositories.

Nanostructured Electrocatalyst for Fuel Cells: Silica Templated Synthesis of Pt/C Composites

94811
C. J. Cornelius

Project Purpose

PtRu blacks are the common methanol oxidation electrocatalysts employed in direct methanol fuel cells (DMFCs). Noble metal utilization in these catalysts has been traditionally low in comparison to anode catalysts used in hydrogen/air fuel cells. One of the reasons for this is the relatively low surface area of these catalysts resulting in inefficient mass transport within the fuel cell catalytic layer.

By structuring the electrocatalyst on the nanoscale, advantageous local mass transfer properties can be attained. The essential balance of kinetic and transport properties of the electrocatalysts are due in part to the hierarchical structure that combines distinct structural considerations across length-scales. This work with the University of New Mexico (UNM) will assist in understanding these properties and develop enhanced materials through the synthesis and investigation of novel bimetallic PtRu nanowire networks.

FY 2006 Accomplishments

The primary accomplishment of the work during FY 2006 was the exploration of the synthesis processes and electrochemical activity of nano-structured Pt-Ru nanowire networks. Because the ability exists to alter and fine-tune the Pt-Ru microstructure, fundamental studies of the local structures can be undertaken next year. By controlling the morphology of the Pt-Ru catalyst on the nanoscale, we can study the effects of the local structure on electrochemical activity and a new class of catalysts will be developed.

We formulated and investigated an innovative aerosol-based synthesis approach that involves using silica precursors and/or preformed particles to template the Pt and Ru precursors, which is followed by silica removal. In this method of materials self-assembly, a solution of metal precursors and the silica template are atomized and undergo spray pyrolysis. This process results in colloidal silica particles in the precursor solution that form a mesostructured Pt-Ru network via the silicate precursors, resulting in a final microporous PtRu network. A combination of variables will be used to generate a hierarchically structured Pt-Ru nanowire network. Confirmatory studies of the structure and morphology are ongoing.

We performed studies of the electrochemical behavior of these catalysts on a rotating disk electrode. Initial studies show that 70 wt.% samples have an order of magnitude increase in activity compared to the initial formulations. We are benchmarking aerosol-derived catalysts with respect to the most advanced industrial catalysts for methanol oxidation, PtRu Vulcan XC72. While the activity per gram of catalyst is not at the level of a commercial catalyst, improvements in aerosol-derived materials suggest its capacity.

Researchers at UNM will continue their effort in developing synthesis methods for high surface-area methanol electro-oxidation catalysts. Optimal formulation of the precursor solution for the aerosol synthesis process will be determined. We will commence model fuel cell investigations in a 5 cm^2 membrane electrode assembly to study the behavior of this new catalyst and to improve the catalytic activity of methanol electo-oxidation catalysts. We will explore promoters such as V and W oxides. These oxides will be included in the sol-gel synthesis route and an integrated synthesis protocol will be developed. We will explore mesoporous materials templating also as a method for nanophase stabilization, and will develop a phenomenological model of catalysis in open frame structured materials.

Significance

An innovative aerosol-based synthesis approach is employed that involves using silica to template Pt and Ru precursors, which is followed by silica removal to form nanostructured catalysts. In this method of assembly, a precursor solution is atomized and undergoes spray pyrolysis. The catalyst precursor solution consists of colloidal silica particles with an average diameter of 20 nm, and metallic-amine platinum and ruthenium complexes. In this method of synthesis, all phases are in intimate contact during synthesis, which promotes the production of a homogeneous material with a higher degree of alloying. The dried powder is then reduced under hydrogen flow at 300 °C for two hours. This is followed by removal of the silica template with a 7M KOH solution.

This research has the potential to access more of the catalytic area than traditional catalysts, thus affording the potential to achieve very low loadings in both methanol and hydrogen fuel cells.

Refereed Communications

E. Switzer, M. Bore, A. Datye, and P. Atanassov, “Hierarchically Structured Platinum-Ruthenium Electrocatalysts for Direct Methanol Fuel Cells,” in *Proceedings of the 209th Electrochemical Society*, p. 1145, May 2006.

Piezoelectric Properties of Arrayed Nanostructures of Zinc Oxide for Sensor Applications

94812
D. Scrymgeour

Project Purpose

The drive toward smaller and more sensitive sensors for hazardous gas, explosive material, and biological agent detection is naturally leading toward the use of nanostructured materials and devices. The advantage of nanostructured sensors is that the small size leads to enhanced surface area to volume ratios ideal for ultrasensitivity.

New semiconductor and oxide materials like zinc oxide (ZnO) that are now being grown at the nanoscale have many exciting cross-coupled properties like the piezoelectric effect that interlink various material states – elastic, optical, and electrical – and provide vast utility to these smart materials when compared to the traditional nanomaterials of silicon and carbon nanotubes. Using piezoelectric nanostructured ZnO, one can create electrically addressable nanoscale mechanical devices. Such structures can provide both actuation and sensing capabilities through the converse and direct piezoelectric responses, respectively.

In this Truman fellowship project, we are characterizing the piezoelectric and electrical properties of ZnO in nanoscale geometries using scanning force microscopy techniques and nanoscale electrical impedance measurements. The basic research provides the groundwork for creating ultrahigh sensitivity sensors that use piezoelectric effect and the resistive and capacitive properties.

Piezoelectric sensors operate by measuring the frequency shift and resonant impedance change of the piezoelectrically generated acoustic waves. The propagation of these waves is strongly dependent upon the material/environment interface and can be shifted by surface-adsorbed species. Additionally, the resistive and capacitive properties of nanostructures are profoundly affected by interaction of the surface with gaseous species. The extremely high surface area to volume ratio, the inherently high resonant frequencies, and the surface-sensitive nature of the electrical properties of these nanostructures will enable the creation of small, accurate, sensors to target specific agents (gas, explosive, biological).

FY 2006 Accomplishments

Piezoelectric force microscope capabilities were constructed and added onto a current scanning force microscope. This technique is used to measure small piezoelectric distortions induced on the sample surface by AC voltage applied to a conductive tip in contact with the sample surface. This capability extends the capabilities of the scanning force setup to determine both the magnitude of the piezoelectric response and orientation of the piezoelectric crystal with resolutions down to tens of nanometers. We investigated various calibration schemes, and calibrated the instrument to allow for quantitative analysis of the piezoelectric effect at nanoscales.

We performed piezoelectric measurements on more than 200 single nanorods and compared results to single-crystal ZnO with known orientation. These results establish that the all the nanorods are [0001] oriented, which contrasts with preliminary morphological examinations of ZnO nanocrystals. We found that the amplitudes varied from rod to rod and are not strongly correlated to the physical dimensions like rod height or radius. The amplitude response was measured at 4.41 pm/V with a standard deviation of 1.73 pm/V, compared to only 2.97 ± 0.57 pm/V for the single crystal.

We performed measurements of the current-voltage characteristics on the nanostructures using conductive atomic force microscopy. Initial experiments show the nanorods create Schottky contacts with the tip. Modeling the response via thermionic emission theory, the ideality factors are in the range 1-4, with barrier heights of 0.70 ± 0.14 eV for diamond coated tips. Correlation of these properties to the measured piezoelectric properties is ongoing.

Significance

The demonstration of piezoelectricity in nanostructures of solution grown ZnO is an experimental first. The combination of this property along with the ease of growth on substrates at temperatures lower than 90 ºC and the previously established ability to pattern the zinc oxide nanocrystals can be used to create macroscale arrays of nanorods of piezoelectric material. This could be used in acoustic band gap crystals, nanoscale elastic wave generators, and nanoscale piezoelectric actuation and transduction.

The integration of nanoscale piezoelectric particles into polymer blends to create piezoelectric nanocomposites has not yet been explored. Ordered arrays of nanorods backfilled with polymer would be a nanoscale analog to 1-3 piezoelectric composites currently used in ultrasonic wave generation. Because the dimensions of the individual piezoelectric rods are very small, the resonant frequency of such an array would have a operating frequency much greater than the MHz regions typically found in traditional 1-3 composites.

Additionally, zinc oxide is a semiconductor with unintentionally carrier concentrations up to ~ 10^{20}. This could be exploited to create devices based on the interaction of piezoelectrically generated acoustic waves with conduction electrons. Similar work using bulk piezoelectrically active semiconductors investigated current oscillations and acoustic amplification in such materials and created acoustic delay lines and acoustic-wave amplifiers. Similar effects and devices have not yet been explored in nanostructured materials.

Refereed Communications

D.A. Scrymgeour and J.W.P. Hsu, “Piezoelectric Properties of Solution Grown ZnO Nanocrystals,” presented at the Materials Research Society Fall 2005 Meeting, Boston, MA, December 2005.

D.A. Scrymgeour, T.L. Sounart, N.C. Simmons, Y.-J. Lee, P.G. Clem, and J.W.P. Hsu, “Piezoelectric and Electrical Properties of Solution Grown ZnO Nanorods,” presented at the Electronic Materials Conference 2006, State College, PA, June 2006.

D.A. Scrymgeour, T.L. Sounart, and J.W.P. Hsu, “Piezoelectric and Electrical Properties of Solution Grown Semiconductor ZnO Nanorods,” presented at the Materials Research Society Spring 2006 Meeting, San Francisco, CA, April 2006.

Three-Dimensional Analysis for Nanoscale Materials Science

94814
I. Arslan

Project Purpose

The major goal of this Truman fellowship project is to study the materials physics of nanomaterials through the development and application of state-of-the-art techniques in electron microscopy in order to understand their structure-property relationships. We will focus on three materials systems: dislocations in thin film p- and n-type GaN, GaN and GaN/AlN core/shell nanowires, and ZnO nanorods interfaced with a polymer. The common theme of these three efforts is to understand how the structure and geometry impact the functionality of the three different semiconductor materials.

First of all, we want to understand why the solid-state lighting semiconductor GaN can be made into devices that function despite the high density of threading dislocations. To solve this, we need to characterize the dislocation on the atomic scale using imaging and spectroscopic techniques. Through a combination of experimental and theoretical work, we are coming to the conclusion that all of the dislocations are controlled by impurity segregation. Not all of the dislocations are deleterious, but if impurities have segregated to a particular dislocation, then the material's properties appear to be altered. Further work needs to be done here, and a comparison between p- and n-doped thin films will be made.

Second, we are working to understand the effect of constraining GaN and its alloys to one dimension, and studying the quantum effects. We plan to measure the band gap of the nanowire at different diameters, as this is very important to precise device fabrication. We expect that the band gap will increase with decreasing nanowire diameter. Also, the material will now be dominated by surface effects, because the surface-to-bulk ratio is high. We want to understand what these effects are, and how they impact device functionality. We will use a combination of atomic, electronic, and three-dimensional (3D) measurements in the electron microscope, as well as conductivity measurements and theoretical calculations.

Lastly, we want to see how the interface affects the properties of the ZnO/polymer functionality. These materials are being developed for solar cell device applications. However, the bonding between the polymer and ZnO is not understood, and the surface reconstruction of the ZnO is also not known. Using high-resolution electron microscope techniques, we will probe the atomic and electronic structure at the interfaces to try to answer some of these questions.

FY 2006 Accomplishments

We spent much of our time on the installation of detectors, hardware, and software. In addition, we performed some preliminary experiments, learned how to operate the complex monochromator that allows the energy resolution (~ 0.1 eV) to measure band gaps, and performed the preliminary measurements of band gaps of GaN nanowires (NWs) at several different diameters. Also with monochromated electron energy loss spectroscopy, we made preliminary measurements of electronic states at the surface of the NWs that show a different signature than bulk.

For core/shell NWs, we analyzed GaN/AlN NWs using high-resolution scanning transmission electron microscopy (STEM) in two dimensions, and found that the GaN inside the AlN is not continuous everywhere, which was a great surprise. We also performed the first STEM tomography experiment to achieve a 3D tomogram of these NWs.

While this provided useful information on the morphology of the outer AlN layer, we were unable to extract information on the inner GaN core. We will now use energy-filtered transmission electron microscopy (EFTEM) tomography to separate the core (Ga signal) and shell (Al signal). We expect EFTEM will allow us to reconstruct the two phases clearly.

We performed the first experiment and reconstruction of a 90° tilt series on a test sample to demonstrate feasibility.

Finally, we performed the preliminary analysis of dislocation cores and found that point defects and intrinsic impurities are responsible for the change in electronic structure at dislocation cores. We will now perform the theoretical calculations to identify the responsible defects.

Significance

GaN and its alloys are an important class of semiconductors being developed for high-power electronics, light emitting diodes (LEDs) and laser diodes in the blue region of the spectrum. The combination of blue LEDs with the existing and optimized red and green LEDs will produce very cheap and efficient white light with long lifetimes. The research described will contribute to realizing this goal. Also, ZnO interfaced with a semiconducting polymer has demonstrated potential as a solar cell device. Therefore, this project will impact the energy mission of DOE.

Tribological Studies of Microelectromechanical Systems

94830
B. R. Antoun

Project Purpose

Polysilicon micromachines have been designed for the purpose of studying the tribological properties of microelectromechanical systems (MEMS). A device for characterizing interfacial adhesion and a device for characterizing friction have been developed at the University of California at Berkeley (UCB). We used these devices to develop an understanding of the physics controlling the interfacial behavior that is critical to the reliability and efficiency of micrometer-scale devices. This research was conducted with Shannon Timpe at UCB.

FY 2006 Accomplishments

Two MEMS tribology testing devices have been successfully designed and implemented, one for testing adhesion alone and a second capable of testing both adhesion and friction. We completed the first experimental phase, which examined the adhesion force under a variety of loading and environmental conditions. In the second experimental phase, we used the second device and attempted to determine the effect of high-adhesive forces on the frictional behavior of the MEMS structures.

Once we established these working devices and experimental protocols, we performed initial experiments on dynamic friction in a variety of environments.

We are conducting experiments to study the effects of current flow across an interface on the adhesive properties of that interface. This is a particularly important topic for microswitching applications. Initial data shows a combination of charge trapping in native oxide films and eventual catastrophic failure due to microwelding.

Significance

This project benefits the general scientific and technical community by developing an understanding of the tribological behavior of MEMS devices under various environments and conditions. This project further serves to develop collaboration with UCB and enable the development of future engineers and scientists that will enter the S&T community and possibly consider employment at Sandia.

Refereed

S.J. Timpe and K. Komvopolous, “An Experimental Study of Sidewall Adhesion in Microelectromechanical Systems,” *Journal of Microelectromechanical Systems*, vol. 14, No. 6, pp. 1356-1363, December 2005.

S. Timpe and K. Komvopolous, “Dynamic Friction in Microelectromechanical Systems,” presented at the Materials Science & Technology 2006 Conference, San Antonio, TX, October 2006.

Tunnel Gap Modulation Spectroscopy: An Ultrasensitive Technique for Measuring Small Mass Change

96088

S. W. Howell

Project Purpose

We are exploring the feasibility of using carbon nanotubes (CNTs) as ultrasensitive mass sensors using the newly developed technique of tunnel gap modulation spectroscopy (TGMS). TGMS accurately measures the natural frequency of oscillation of a single CNT at room temperature by monitoring the electron tunnel current between the CNT and a conducting substrate. Since the magnitude of the tunnel current is exponentially dependent on the CNT-substrate separation, small changes in the CNT's motion can easily be detected. Our collaborators in the Reifenberger Nanophysics Lab at Purdue University demonstrated the feasibility of using TGMS as a novel nanoscale oscillator that detects a radio frequency (RF) signal derived from the tunnel current of an oscillating CNT.

To develop chemical vapor sensors, we will functionalize individual CNTs with Sandia's unique enabling diazonium/iodonium sensor chemistries that will selectively bind various analytes. When ultralow amounts (< 1 fg) of a target analyte bind to a functionalized CNT, the CNT's natural frequency of oscillation will dramatically shift due to the CNT's small mass (< 1 pg). As an example, we estimate that using a functionalized CNT, a frequency shift of 0.57 MHz will result when a single virus (10-15 g) is attached to the end.

Although initial experiments will be conducted using multiwalled carbon nanotubes (MWNTs), the method seems generally applicable to most nanowires. Successful completion of this project will enhance Sandia's ongoing nanosensing programs by enabling fundamental research in carbon nanotube-based sensing and device development.

FY 2006 Accomplishments

In FY 2006, we obtained atomic resolution scans of highly orientated pyrolytic graphite with a scanning probe microscope. These atomistic scans provide a stability benchmark to evaluate the resolution of Purdue's custom RF-STM head.

We refined the experimental apparatus needed for tunnel gap modulation spectroscopy. We will conduct initial TGMS measurements by attaching a MWCNT to an STM tip and holding the MWCNT within tunneling range (~ 1 nm) of a conducting substrate. The RF signal (10s of MHz) generated by the thermally-excited vibrating MWNT will be amplified and detected with a spectrum analyzer. A custom RF-STM amplifier will be used to measure the tunnel current.

We also modeled the mechanical behavior of the nanotube tip by:

- Calculating the oscillation amplitude, frequency, and mode shapes of a carbon nanotube using the Euler-Bernoulli equation and the equipartion theorem.
- Using ANSYS, a finite element analysis program, to model the CNT as a cantilevered beam. Point masses added to the CNT simulated the presence of a bonded analyte.
- Using Matlab to calculate the frequency spectra of the oscillating MWNT's tunnel current.

We used a circuit modeling program, ORCAD Capture, to design a custom RF-STM head. ORCAD simulations of the hybrid RF-STM preamplifier showed that the gain of the high-frequency converter is 10^5 (1-100 MHz range) while the gain of the low-frequency (tunnel current-to-voltage) converter is 10^8. STM scans of step edges on graphite show the custom RF-STM head has a fast and stable response and the tunnel current-to-voltage converter has a gain of 10^8. We designed an oscillator circuit that uses a quartz transducer as a 5 MHz reference to test the high-frequency amplifier.

We improved techniques to attach MWNTs to STM tips by refining the procedures to etch nickel STM tips. MWNTs were attached to Ni STM tips using nanomanipulators. Initial tests show the MWNTs are robustly attached to the Ni STM tips. We characterized these tips using a scanning electron microscope.

Significance

In order to increase the sensitivity of cantilevered microsensors and nanosensors, the mass of the cantilever needs to be decreased. MWNTs are ideal cantilevered nanosensors because of their small mass (~ 1 picogram) and high quality factor (Q ~ 500). Even the smallest amount of analyte bonded to a MWNT will cause a detectable shift in the MWNT's oscillation frequency.

With the unique enabling diazonium/iodonium sensor chemistry developed by Sandia, MWNTs will be functionalized to detect the presence of chemical warfare (CW) stimulants and toxic industrial compounds (TICs). Once we demonstrate the reliability of measurements of the oscillation frequency of MWNTs, the MWNTs will be functionalized for use as nanoscale chemical and biological sensors.

In addition to their improved sensitivity, cantilever nanoscale sensors offer a low-power (microwatts) sensing platform. Sandia's nonproliferation mission area, specifically monitoring and detection of chemicals, will benefit from the sensitivity and low-power consumption of these MWNT-based sensors.

Other Communications

L. Biedermann, C. Lan, R. Reifenberger, and J. Therrien, "Tunnel Gap Modulation Spectroscopy: A Scanning Probe High-Frequency Oscillator," in *Proceedings of the 6th IEEE Conference on Nanotechnology*, p. 06TH8861C, June 2006.

Optical Properties of Plasmonic Metal-Dielectric Composites

96299
A. V. Smith

Project Purpose

Our project is applying plasmonic resonances of small metal particles to enhance stimulated Raman scattering and thus improve the sensitivity of Raman spectroscopy. The fabrication of thin films containing the particles will be optimized for this application. We are also exploring the application of plasmonic resonance to developing negative refractive index material. Such material might be useful in subwavelength resolution near-field imaging. This project is a collaboration with Mark D. Thoreson, a student at Purdue University.

FY 2006 Accomplishments

We studied semicontinuous metal/dielectric composites for use as infrared obscurants and the effect of different dielectric sublayers on the characteristics of random metal/dielectric films. Subsequent photomodification of these films showed it is possible to create a controlled transparency window in these plasmonic structures. Our goal is to increase the selectivity and spectral shape of these transparency windows.

Previously, we studied various dielectric sublayers, including oxides of silicon, aluminum and titanium. As expected, the dielectric sublayer material affects the structure of the subsequent evaporated metal film. We varied the metal layer thicknesses to match the plasmon absorption characteristics across differing film materials. Subsequent photomodification then produced windows of transparency. We found that samples with a silicon dioxide sublayer proved to be most useful for photomodification, as other sublayer materials were altered by the photomodification process.

We studied semicontinuous metal/dielectric films for use as surface enhanced Raman scattering (SERS) substrates. The first commercially produced prototypes were fabricated from a process developed at Purdue. These prototypes were characterized using scanning electron microscopy, spectrophotometry, and other methods. The commercially fabricated substrates show promise as nanoplasmonic structures. Obtaining large quantities of high quality SERS substrates will allow the study of the time dynamics of the adaptation process these substrates undergo during analyte deposition.

Studies of negative refractive index in semicontinuous films continue. Preliminary simulation results indicate the possibility of incorporating semicontinuous metal/dielectric films into more complex structures to create conditions for negative refraction at optical wavelengths. Initial experimental results indicate that composites made from multiple thin metal/dielectric layers show appropriate surface roughness for use in negative index studies, and might be useful for imaging with super-resolution near-field lenses.

Significance

We showed that photomodification of evaporated, semicontinuous metal/dielectric films can produce spectral transparence windows. This might have applications in tunable infrared filters. We developed prototype metal/dielectric films for use as surface enhanced Raman substrates that are being developed for commercial production through industry/university collaborations. If negative refractive index films can be developed using the techniques under study, such films might be applied to super-resolution imaging in submicron lithography.

Refereed

V.P. Drachev, V.C. Nashine, M.D. Thoreson, D. Ben-Amotz, V.J. Davisson, and V.M. Shalaev, “Adaptive Silver Films for Detection of Antibody-Antigen Binding,” *Langmuir*, vol. 21, pp. 8368-8373, August 2005.

Other Communications

V.M. Shalaev, V.P. Drachev, V. Nashine, M.D. Thoreson, E.N. Khaliullin, D. Ben-Amotz, and V.J Davisson, “Adaptive Silver Films for Bio-Array Applications,” presented at PITTCON 2005, Orlando, FL, February 2006.

Membrane-Based Water Purification for Removal of Arsenic and Biologically Active Small Molecules

97982

M. A. Hickner

Project Purpose

This goal of this project with the University of Texas at Austin was to define some of the unique transport relationships of Sandia membranes and determine their potential for water purification.

FY 2006 Accomplishments

A three-cell cross-flow apparatus was designed and built at Sandia to enhance our membrane characterization capabilities.

Significance

Current commercial membranes for water treatment, mainly composed of cross-linked aromatic polyamides, are improving at the rate of 1-2 percent per year. This improvement is not great enough to spark a revolutionary change in the water treatment industry. Sandia has excellent polymer synthesis and characterization capabilities and has brought these resources to bear on problems such as fuel cells and gas separation membranes.

We are beginning to apply our materials knowledge to water treatment and this LDRD project is in support of that goal. Our key R&D accomplishments provide a new set of water treatment membrane materials, and application-specific testing capabilities for the general S&T community. Our results will benefit Sandia's mission areas in energy and water security.

Microfabricated Preconcentrator for Microscale Gas Chromatography

98104

A. Robinson

Project Purpose

Cryogenic cooling for sample injection is ubiquitous in large-scale commercial gas chromatography (GC) systems. However for microscale systems, preconcentration via thermal desorption is a superior sample injection solution. The goal of this project is to implement a microscale preconcentrator for use as the analytical front-end of a microGC where the primary application is rapid, complex mixture analysis.

At the University of Michigan's Center for Wireless Integrated MicroSystems (WIMS), a microfabricated preconcentrator has been developed to enable high-resolution chromatography with low-parts-per-billion (ppb) sensitivity. The packaging and integration goals for this project include modularity and environmental robustness for reliable integration into a common microfluidic/electrical substrate.

Miniaturization of preconcentrators using packed beds of granular adsorbents for microscale and portable GC systems has not been developed widely due to the difficulty in manipulating micron-diameter granular adsorbents to construct microscale packed beds. The packed bed approach is different from microhotplate-based preconcentrator designs and allows for the accommodation of adequate adsorbent mass to achieve quantitative trapping of trace level volatile organic compound (VOC) mixtures at ppb concentrations for indoor air analysis.

A cavity microheater-based preconcentrator design is used in conjunction with an adsorbent-solvent filling technique and subsequent solvent-compatible wafer-level sealing technology to fabricate preconcentrators capable of quantitative capture of ppb-level VOCs. The preconcentrator design goals are to minimize total analysis time by reducing the preconcentrator sampling times and to reduce the desorbed injection plug width to the microGC column at low column inlet velocities.

Further preconcentrator miniaturization, wafer-level packaging, gas interconnections, and electrical integration with the WIMS microGC are also addressed. Advances in thermal isolation, vacuum packaging, and leak-proof fluid interconnection technologies for the preconcentrator developed in this project benefit other microscale microGC components (columns, substrate, sensor arrays) and portable chemical and biological analysis systems.

FY 2006 Accomplishments

- We built a new preconcentrator measurement system to characterize device performance, including sample extraction and sample injection.
- We demonstrated large adsorptive capacity with our microdevice, including 300 mL exhaustive extraction of octane at 25 mL/minute, room temperature, and parts-per-million concentrations.
- The new effluent curve data are being used in conjunction with a new preconcentrator model to describe the performance limits of packed bed designs.
- Sample injection studies of the micropreconcentrator show injection plug widths on the order of seconds, appropriate for injection onto standard-bore 3 m microcolumns.
- We customized two commercially available packages for use with the preconcentrator to integrate it with macro- and microscale gas chromatography systems. The technology is designed to allow reuse so that a preconcentrator can be tested in a laboratory-scale system for functional verification, followed by microscale integration.

Significance

A combined sample extraction and injection device is key to any real-time gas analysis system, including those for chemical and biological (C/B) warfare agent detection. The microscale preconcentrator results of this project are applicable to the development of these miniaturized C/B detection systems. Key technologies include robust microdevices and electrical interconnects, silicon micromachining techniques, thermal isolation structures and methods, and methods of integration into analytical systems. These leverage Sandia's microsystem efforts.

Results from this research could find use in gas refineries, pharmaceutical plants, chemical factories, semiconductor manufacture facilities, and so on. Gas chromatographic breath analysis for medical diagnosis is another key area presently receiving attention. These and other applications would give this research project impact in defense, energy, nonproliferation, and homeland security.

Dynamics of Propagating Shock Waves and Phase Fronts

98105
J. M. Redmond

Project Purpose

Phase transformations and shocks are developed in many high-energy impact and explosion problems. Understanding the physics of these phenomena in a wide range of materials permits their use in structures designed to mitigate the effects of blast or impact events. This research with the University of Texas at Austin involves a combination of experimental observations, analytical modeling, and numerical simulations of models to examine impacts that generate a fan of elastic waves followed by phase transformation and/or shock wave.

FY 2006 Accomplishments

I. Experimental

- We successfully modified a tensile impact experimental setup to allow visual inspection of a specimen during impact and to allow the capability to increase impact velocity.
- We ran a series of impact experiments on rubber specimens in the modified tensile impact setup, as well as a series of retraction experiments with the use of a high-speed camera to monitor specimen displacement as a function of time.
- We developed a working code that calculates the strain and particle velocity of the rubber as a function of time using images taken during the experiment of lines drawn on the specimen. This code also estimates the constitutive behavior of and the wave speeds in the material during experiment.
- We obtained NiTi specimens and found the heat treatment that allowed the specimens to undergo the phase transformation from austenite to martensite at room temperature. This eliminates the need for heating devices that obstruct the field of view of a high-speed camera.
- We developed a working code that uses the digital image correlation technique to record the strain history of a specimen during loading, and we verified its capability and resolution in quasistatic tensile tests on NiTi.

II. Comparison of Theory and Experiments

- We modified theory developed by Knowles to account for the material properties of the rubber used in the impact and retraction experiments.
- We showed that the theory qualitatively describes the response of rubber to impact and retraction during the elastic fan of waves prior to the arrival of a shock wave, but not quantitatively.
- We showed that during these experiments a partial phase transformation from amorphous to crystalline (impact) or crystalline to amorphous state (retraction) occurs prior to arrival of a shock wave.

Significance

The partial phase transformation of rubber prior to arrival of a shock wave during impact and retraction is a new observation and gives an insight into how to better model the response of rubber. The tensile impact experimental setup and the two codes that can be used to determine strain and particle velocity history provide a new set of tools that can be used to investigate other materials that experience a phase transformation and/or shock wave during impact.

With this new tool set, better models of the kinetic relation can now be developed to fully describe a material's response to a given impact. Knowing the magnitude of the driving force and the speed of the phase transformation front/shock wave, engineers will know when to use these materials and how to design a structure utilizing their material properties for use against certain types of impact.

The accomplishments of this project are beneficial to the scientific and technical communities and to Sandia mission areas in science, homeland security and nuclear weapons. This work will advance the ability to develop accurate constitutive models that predict the mechanical response of materials to extreme conditions and new advanced computational mechanics codes to simulate the response of nuclear weapon components.

Refereed Communications

J. Niemczura and K. Ravi-Chandar, "Dynamics of Propagating Phase Boundaries in NiTi," *Journal of the Mechanics and Physics of Solids*, vol. 54, pp. 2136-2161, October 2006.

J. Niemczura and K. Ravi-Chandar, "On the Propagation of Finite Deformation Waves in Rubber," submitted to *Journal of the Mechanics and Physics of Solids*.

Pareto Optimization Techniques

105185
C. R. Lawton, G. K. Kao

Project Purpose

Multiobjective optimization presents a number of significant technical challenges. The first challenge occurs when conflicting multiple objectives lead to multiple optimal solutions. Second, given that multiple optimal solutions are inherent in multiobjective optimization, evaluating solutions will be significantly more difficult than single objective optimization problems, requiring the need for additional post-optimality analysis of the solution space.

The research focus of this LDRD project can be summarized into two parts. The first part of the research consisted of investigating multiobjective optimization algorithms in the field of reliability analysis. The second part focused on post-optimality analysis, which is the analysis of solutions obtained after the optimization process. These two areas are integral parts of the general multiobjective optimization analysis framework. The project team includes a doctoral student at the University of Illinois.

FY 2006 Accomplishments

We studied several different multiobjective optimization algorithms and applied them to different reliability test problems, including those similar to the analysis of the Apache helicopters and the Naval Landing Craft Air Cushions.

In particular, the Nondominated Sorting Genetic Algorithm II (NSGA-II) was compared to other single and multiobjective genetic algorithms. NSGA-II was shown to be superior in the reliability analysis domain. It was able to efficiently generate large diverse sets of Pareto optimal solutions. Finding large sets of diverse Pareto optimal solutions provides the decision makers with a full spectrum of trade-off analyses. In addition, unlike other multiobjective genetic algorithms (MOGA), NSGA-II requires minimal problem dependent adjustments, using only a few user defined parameters. We are incorporating such multiobjective optimization capabilities into Pro-Opta, a reliability and optimization analysis tool developed at Sandia.

One important objective is helping the decision makers visualize large sets of Pareto optimal solutions. In large and high-dimensional problems, this is not a trivial task. We began investigating interactive approaches to presenting the solutions to the decision maker. This work is also being incorporated into Pro-Opta.

Another important focus is the analysis of the Pareto optimal solutions to enable the decision maker to identify the best solutions across a very large number of Pareto optimal solutions. We focused on finding preferred subsets of Pareto optimal solutions. To do so, we introduced and formulated a new discrete optimization problem to obtain such subsets. We compared and studied two exact algorithms and five heuristics.

We are also trying to identify characteristics of Pareto optimal solutions. Such characteristics can help identify solutions that are robust and desirable. Several evaluation metrics were proposed and are under investigation. Other techniques such as clustering are also under evaluation. By gaining more information about Pareto optimal solutions, we expect to acheive overall improvements in the multiobjective optimization process.

Significance

The area of multiobjective optimization presents a number of significant technical challenges; specifically, when conflicting multiple objectives lead to multiple optimal solutions. That is, it is very difficult to find a set of well-distributed optimal solutions that can be evaluated by the analyst within the context of their domain-specific problem. This in turn leads to the need for visualizing complex multiobjective Pareto fronts.

As Sandia extends its GO genetic optimization software into the multiobjective realm, capabilities developed in this project will contribute to the development of a leading multiobjective optimization tool that will provide significant technical benefit to Sandia's nuclear weapons, homeland security, and defense missions.

Refereed Communications

C.R. Lawton and G.K. Kao, "Finding Preferred Subsets of Pareto Optimal Solutions," to be published in the *Journal of Computational Optimization and Applications (COAP)*.

An Examination into the Chemical Properties of Supercritical Water

105187
J. V. Zuffranieri

Project Purpose

This project with the University of Wisconsin is designed to establish concentrations or G-values of radicals formed per energy ("G-value" usually suggests molecules per 100 eV) for both neutron and gamma radiation as a function of temperature and pressure up to and beyond the critical point. Two separate pieces of data are needed to properly understand and simulate water chemistry at a given temperature and pressure. First, it must be known what concentration of radicals will form based on the dose from high and low linear energy transfer radiation. Second, the recombination rates of these radicals must be known.

In addition to direct water chemistry data, critical hydrogen concentration experiments are also run. These determine the amount of hydrogen necessary to sufficiently prevent the formation of all oxidizing species. Such water chemistry information will prove useful and will aid in the design and performance of Generation IV reactors.

FY 2006 Accomplishments

One of the challenges we met was the difficulty in measuring neutron and gamma dose to the water in the irradiation volume. Gamma dose was particularly difficult to measure since instruments and foils typically used to measure such high doses see interference from the high neutron dose.

To overcome this challenge, we designed a series of experiments: 1) a neutron activation experiment using a sodium carbonate solution to determine thermal neutron flux; 2) a Monte Carlo N-Particle model of the reactor to determine neutron energy deposition as a function of thermal flux; 3) a radiolysis experiment measuring the concentrations of aqueous electrons and hydrogen radicals as well as the long-term formation of hydrogen gas at 25 °C or 100 °C; and 4) a simulation using known values at these temperatures to calculate the G-values for these radicals by both neutron and gamma radiation. Experiments 1 and 2 can be used to determine total dose, experiment 3 finds total concentrations produced, 4 finds expected concentration from neutron dose, which can be subtracted from 3, thereby leaving gamma produced concentration, which can be translated into dose again by 4.

Significance

The supercritical water reactor, one of the Generation IV reactor designs chosen for research and development in the US, faces many challenges in the design of materials that will be in contact with the water, especially at the core outlet. Chief issues already addressed in pressurized water reactors are worsened by higher pressure and temperatures as well as additional factors caused by the extreme changes in water properties at the critical point.

Water chemistry will play a major role in the feasibility of using different types of steels, nickel-based alloys, and other metals as fuel cladding, pressure vessel lining, and piping of the system. Water radiolysis, which can create oxidizing species and change the corrosion potential of the cooling water, will be a significant aspect in consideration of water chemistry.

The results garnered from these experiments will be helpful in bringing about the implementation of the Generation IV reactors in the US, which will play an important part in defining the role nuclear energy will have in America's future.

Data Collecting, Analysis, and Modeling to Better Understand Supercritical Water (SCW) Reactor Safety Technologies

105188
J. V. Zuffranieri

Project Purpose

In order to advance the development of supercritical water (SCW) reactors, mechanisms of heat transfer to supercritical fluids need to be further clarified to improve heat transfer calculations. Currently, there are no detailed velocity, density, or turbulence profiles obtained in water at critical pressures. The purpose of this experimental investigation with the University of Wisconsin is to develop a set of heat transfer measurements along with detailed information on the fluctuating velocities and density, in addition to the thickness of the momentum and thermal boundary layers. The experimental results will serve as a database to compare existing models and aid in the development of new models that will be used to improve performance and decrease cost by increasing the efficiency of the systems under investigation.

FY 2006 Accomplishments

We built a SCW heat transfer test facility to allow for a detailed study of heat transfer to SCW in a circular annular geometry. The loop has dimensions of approximately 2 m wide by 3 m tall and is made of 4.29 cm inner diameter Inconel 625 piping. A 3.3 m long heater rod with a diameter of 1.07 cm spans the entire right leg of the loop and protrudes out both ends.

This design permits the use of 16 thermocouples evenly spaced along the inner cladding of the 1 m heated length. The center portion of the right leg of the loop serves as the test section, allowing a 76 cm entrance length for both upward and downward flow studies. The heater can generate up to 50 kW, giving a maximum heat flux of 1.5 MW/m^2. A pump capable of operating at supercritical conditions generates mass velocities in the range of 200 to 2000 kg/m^{2}s. The current configuration is up-flow, although the facility can be used for flows in either direction with only minor modification. The setup is capable of operating at any steady-state heat flux condition by using a variable heat removal system made up of copper cooling coils. Eight copper coils of various contact area are tightly wrapped to the Inconel piping. Heat removal by the cooling coils can be set to match that supplied by the heater by simply controlling which coils receive cooling water and controlling the respective flow rates.

About 33 percent of the heat transfer measurements for the planned test matrix have been completed. These experiments have been performed at a pressure of 25 MPa, with bulk inlet temperatures of 300 °C to 395 °C, heat flux of 250 kW/m^2 to 1MW/m^2, and mass velocities of 400 kg/m^{2}s to 1400 kg/m^{2}s.

Significance

In an effort to improve the efficiency of current light water reactors (LWRs), the Generation IV initiative has included the SCW reactor as one of the next steps in future nuclear reactors. A SCW reactor will achieve efficiencies of about 45-50 percent, compared with current LWR efficiencies of about 33 percent, by operating its coolant at higher temperature (500 °C) and pressure (25 MPa) than current LWRs. These operating conditions are above the pseudocritical temperature of water (defined as the temperature, for a given pressure, at which the specific heat exhibits a maximum) and thus the coolant of SCW reactors will undergo large thermophysical property changes.

Current models and correlations used for water near or at the supercritical region have not been able to accurately predict experimentally measured heat transfer coefficients, which has negative effects on system performance. With the Generation IV reactors set to come online in the near future, it is important to have a better understanding of the properties related to SCW reactor performance. This work serves a vital need in improving this understanding.

Unpublished Projects

For information on the following FY 2006 LDRD projects, please contact the LDRD Office:

Laboratory Directed Research & Development
Sandia National Laboratories
Albuquerque, NM 87185-0123

Project Number	Title
93419	Advanced Surety Concepts
67087	Infrastructural Development for Flexible Network of Devices
93625	Strategic Concepts for Information Superiority
93626	Ultralow Power Management Circuit Design
93634	Binary Analysis for Embedded Environment
93635	Software Evaluation in Virtualized Environment
104513	Run-Time Reusable Software Components
104576	Image Processing Software for Reverse Engineering Applications
105189	Precise Distributed Control and State/Parameter Estimation for Multibody Satellites and Satellite Formations
105190	Modeling and Design of Microstructures with Tailored Adhesive Properties
105191	Fourier Analysis and Synthesis Tomography
105193	Neural Correlates of Attention and Intention in Decision Making of Macaques and Humans: Selective Lesioning of Posterior Parietal Areas during Electrophysiology and fMRI
105213	Application of Advanced Laser Diagnostics to Hypersonic Wind Tunnels and Combustion Systems
105672	On the Role of Numerical Error in Turbulence Simulations

Appendix A: Awards/Recognition List

Description	LDRD Project Title
R&D 100 Award, R&D Magazine	• Algorithmic Support for Commodity-Based Parallel Computing Systems (project 26516)
C. Jeffrey Brinker named Regents' Professor at the University of New Mexico	• Superhydrophobic Surface Coatings for Microfluidics and MEMS (project 73185) • Exploiting Interfacial Water Properties for Desalination and Purification Applications (project 90493)
C. Jeffrey Brinker received a Distinguished Alumnus Award from Rutgers University	• Superhydrophobic Surface Coatings for Microfluidics and MEMS (project 73185) • Exploiting Interfacial Water Properties for Desalination and Purification Applications (project 90493)
Chris Cornelius received the American Indian Science and Engineering Society Technical Excellence Award	• Development of a Universal Fuel Processor (project 90497) • Creation of Water-Treatment Membrane Technologies with Reduced Biofouling (project 102737)
Rafael Davalos received the HENAAC National Award for Most Promising Engineer or Scientist	• Precisely Controlled Picoliter Vessels with Rapid Sample Preparation for Trace Biotoxin Detection (project 67085)
Randall T. Cygan named Fellow of the Mineralogical Society of America	• Exploiting Interfacial Water Properties for Desalination and Purification Applications (project 90493)
ASME Solar 2006 Best Paper Award for outstanding papers in Solar Chemistry and Hydrogen Technologies, July 2006. Four papers representing the work from this LDRD were collectively recognized.	• Innovative Solar Thermochemical Water Splitting (project 79781)
Best Paper Award, Materials Research Society Symposium, November 2005	• Noncontact Surface Thermometry for Microsystems (project 67067)
Adrian Rodriguez, a University of New Mexico undergraduate student, won first prize in the poster competition at the 42nd Annual Symposium/New Mexico Chapter of the American Vacuum Society.	• Engineered Conjugated Molecule-Linked Metal Nanocrystal/Silica Arrays for Integrated Chemical Sensor Platforms (project 102599)

Appendix B: Project Performance Measures

Refereed Publications	**389**
Other Communications	**360**
Technical Advances	**128**
Patent Applications	**37**
Post-Doctoral Researchers	**86**
New Hires	**6**
Awards	**9**

Appendix C: Mission Technology Areas

Science of Complex Systems	**148**
Modeling and Simulation of Complex Systems	**160**
High-Performance Computing	**69**
Nuclear Weapons Predictive Capability (Classified)	**6**
Safety Assessment and Engineering	**20**
Use Control and Security	**53**
Nonproliferation and Verification	**136**
Dynamic Testing in Severe Environments	**24**
High Energy Density Materials	**21**
Laser Technology	**33**
Pulsed Power and Accelerators	**19**
Microelectronics and Photonics	**166**
Nuclear Materials	**12**
Engineered Materials	**141**
Flexible Manufacturing	**46**
Environmentally Conscious Manufacturing	**7**
Intelligent Machines and Robotics	**56**
Waste Management	**17**
Other	**63**

Appendix D: Major National Programs

Waste Management	**23**	Department of Energy
Energy Efficiency & Renewable Energy	**87**	
ES&H	**8**	
Environmental Restoration	**25**	
Fossil Energy	**24**	
Civilian Radioactive-Waste Management	**13**	
Economic Impact	**46**	
Energy Research	**85**	
Intelligence & National Security	**202**	
Nuclear Energy	**28**	
Science Education and Technical Information	**60**	
Defense Programs	**262**	
Department of Defense	**218**	Work for Others
Other Federal Agencies	**54**	
Other (Industry, Consortia…)	**28**	
Department of Homeland Security	**205**	

Appendix E: Capability Areas

Capability Areas	Number of Projects
Aeronautics	**39**
Applied Molecular Biology	**53**
Ceramics	**34**
Composites	**27**
Computer Simulation and Modeling	**147**
Data Storage and Peripherals	**9**
Electronics and Photonics	**148**
Energy	**83**
Flexible Computer-Integrated Manufacturing	**18**
High-Definition Imaging and Displays	**24**
High-Performance Computing and Networking	**49**
High-Performance Metals and Alloys	**16**
Intelligent Processing Equipment	**29**
Material Synthesis and Processing	**97**
Medical Technology	**61**
Micro- and Nanofabrication	**176**
Micro- and Optoelectronics	**126**
Photonic Materials	**51**
Pollution Minimization, Remediation, and Waste Mgt.	**28**
Sensors and Signal Processing	**186**
Software	**63**
Surface Transportation Technologies	**13**
Systems Management Technologies	**33**