# LDRD 2005

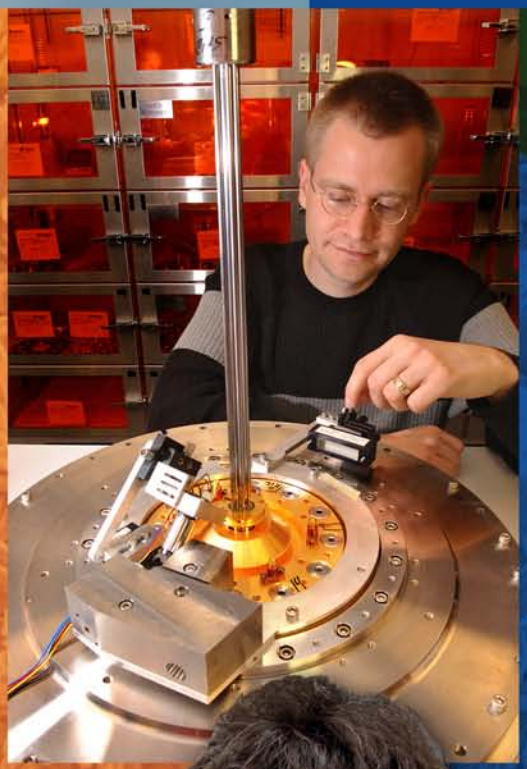
## LABORATORY DIRECTED RESEARCH AND DEVELOPMENT

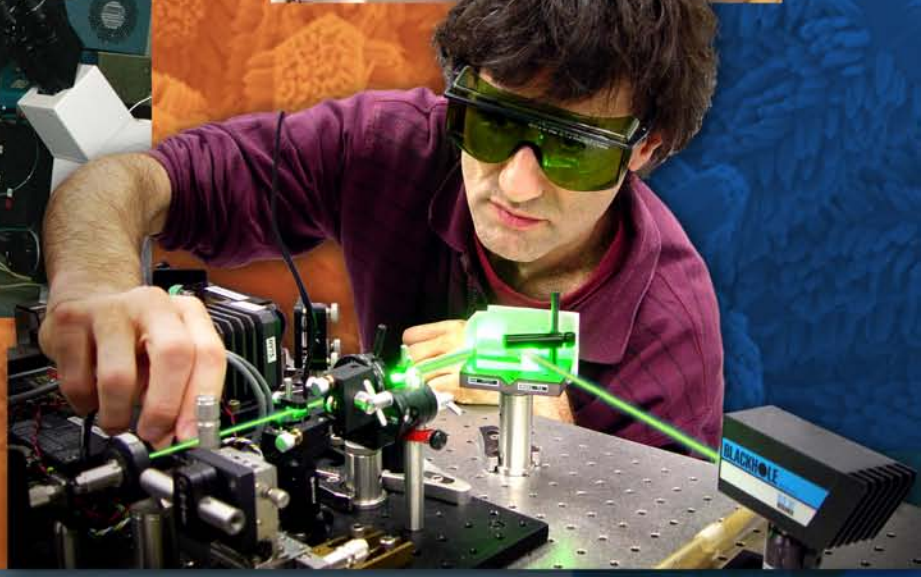
Science, Technology, and Engineering

Mission Technologies

Corporate Objectives

Grand Challenges





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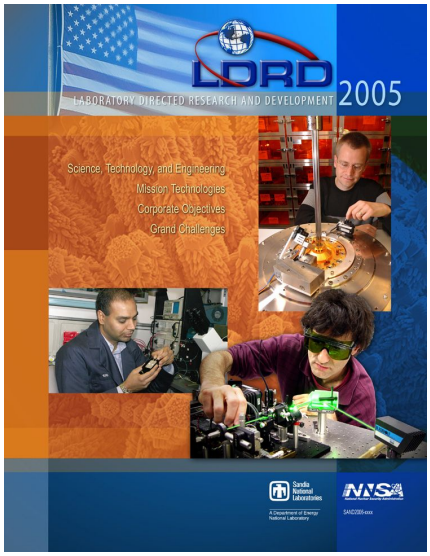


Cover photos (top right going clockwise):
*(1) Sandia Researcher Daniel Sinars demonstrates the setup he and his team created to peer into the center of Sandia's Z machine at the moment of firing. The crystal under his finger is attached to portions of a Z target configuration.*

*(2) Sandia Researcher Dahv Kliner demonstrates a bend-loss-induced mode filtering method that allows the use of large core diameter fibers while maintaining the high beam quality.*

*(3) Sandia Researcher Anup Singh inserts an immunoassay chip into the microfluidic module of the hand-held device.*

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## Abstract

This report summarizes progress from the Laboratory Directed Research and Development (LDRD) program during fiscal year 2005. In addition to a programmatic and financial overview, the report includes progress reports from 410 individual R&D projects in 19 categories.

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*LDRD Annual Report Staff:*

Hank Westrich
Donna Chavez
Keith Ortiz
Danielle Montoya
Sheryl Martinez

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# An Overview of Sandia's FY 2005 Laboratory Directed Research and Development (LDRD) Program

*K. Ortiz and D. Montoya*

> ***Aviation Week & Space Technology***
> **The Right Stuff**[1]
> To attract best talent, survey says aerospace/defense contractors must focus on stimulating jobs and product development
>
> *Aerospace/defense contractors committed to paying more than lip service to attracting the best and brightest talent need to concentrate on two imperatives: technologically challenging jobs and a robust product-development pipeline.*
>
> *Sandia National Laboratories, an Energy Dept. research facility managed by Lockheed Martin Corp., ranked highest for the second consecutive year in terms of providing technological challenges, valuing its employees and promoting diversity in the workplace....*

## The Role of LDRD in Providing Technological Challenge and Innovative Products

Sandia is well known for its technologically challenging work and innovative product development performed in service to national security. Beginning with a base of work to support nuclear weapons, Sandia has grown to serve a broad spectrum of national security customers, including the military, nuclear nonproliferation, intelligence, energy, and homeland security. Technological challenge and innovative product development for these customers are inherent to the success of the Laboratories and critical to achieving sustainable national security.

LDRD projects hold an especially valuable place in Sandia's portfolio of work. LDRD is the Labs' sole source of discretionary funding and represents less than 6% of the Labs' total budget. LDRD funding is used to solicit R&D that is not specifically tied to ongoing program spending. This allows the LDRD Program the unique flexibility to quickly respond to newly perceived needs and opportunities and to explore disruptive and revolutionary science and technology that anticipate future mission space and future R&D needs. LDRD projects represent some of the most advanced, highest risk, and potentially highest value research and development. These are challenging projects that are still too risky and future driven for our customers to fund.

Consider project number 55086, "Prompt Global Response." This project investigates technologies necessary for long range, extended duration hypersonic flight. The idea for this project came from reading the 2001 *Quadrennial Defense Review* and *Nuclear Posture Review* (released in September 2001 and December 2001); the proposal was written and reviewed in about six months, and the project started at the beginning of FY 2003 (October 2002). The technical risks of this project were far too high for it to be funded by the government or industry. Yet its potential value to Sandia and the nation is enormous: the project's vision is to enable a rapid military response to a threat anywhere around the globe. Researchers identified promising candidate high temperature materials; improved high Mach number aerodynamic codes; and developed high speed navigation, guidance, and control technologies. The project is generating considerable excitement by pushing the forefront of hypersonic flight. In the words of one of the project's technical reviewers, "This is what top aerospace engineers want to do." Indeed, this is exactly what *Aviation Week & Space Technology* means by "technologically challenging."

Regarding "a robust product-development pipeline," the innovation pipeline at Sandia is often primed by LDRD. LDRD projects are always relevant to our national security missions and often have in mind a particular impact on the product development pipeline. However, most LDRD projects have multiple potential benefits and should not be thought of as having a single assignable benefit.

For example, project number 67026, "Development of GaN 20-100 Watt Ku-Band Power Amplifiers for MicroSAR," is developing high power, solid state gallium nitride devices as candidates to replace the

1. C. Hedden, "The Right Stuff," *Aviation Week & Space Technology*, www.aviationnow.com/awst, September 5, 2005.

traveling-wave tubes currently used in synthetic aperture radar (SAR). This replacement could result in a large reduction of SAR system size and cost. Together with other LDRD efforts, this would enable SAR systems to fly on much smaller platforms, e.g., tactical unmanned aerial vehicles (UAVs), thereby opening the benefits of SAR technology to all our national security customers. To accomplish the intended product development goal, basic problems associated with gallium nitride devices must be solved in thermal management, device characterization, device stabilization, power combining, and packaging. Solving these basic problems for this device gives Sandia the capability to innovate with gallium nitride for numerous other applications. The benefits of this project transcend its title and go beyond synthetic aperture radar and beyond high power, high frequency power amplifiers.

Another project, number 81752, "Integrated Fiber Lasers for Efficient High Power Generation," is increasing the power output from fiber lasers to over 1 MW for high power applications. It also has a goal of integrating nearly all of a high power laser system's components into a rugged, monolithic fiber-optic platform. This approach is analogous to the integration that has occurred in microelectronics, microfluidics, and microelectromechanical systems. This may create a revolution in laser system architecture resulting from the greater efficiency of fiber lasers and from the integrated package. High power fiber laser systems are of known value to many national security applications, such as military applications, laser communications, and laser detection and interrogation of chemical plumes. The compact size and ruggedness of integrated fiber laser systems will also create opportunities for many other applications. Again, through innovative technology development, the benefits of the project go beyond its title and beyond the immediate applications that require high power.

It is too soon to predict exactly what innovative products will be created with these technologies. They have already attracted the attention of our customers and other staff eager to take advantage of the new technology. LDRD is priming the pipeline.

One more reason that LDRD projects are especially valuable is because they attract a disproportionate amount of media and professional attention, both inside and outside the Labs. LDRD projects produce conference papers and technical journal articles at a higher rate than other work. LDRD projects are often the subject of media releases from Sandia that are picked up by newspapers and magazines. For example, recent television coverage and a prominent article[1] in the *San Francisco Chronicle* recently described the efforts of project number 55087, "Winning the War: A Systems Approach to Defending Our Borders." Thus, LDRD is playing a role in shaping the public's idea of Sandia and the first impressions of the scientists and engineers we want to attract.

The role of technologically challenging work and innovative product development was summarized by then Laboratories' Director, C. Paul Robinson, for the 2004 Aviation Week & Space Technology annual workplace survey:[2]

> *"People want to be part of the lab. When you have the really hardest problems, you send them here to be solved. That challenge is what brings people here. Once they get a steady diet of it, it's pretty hard to get anywhere else . . . [Plus], it's not just a job. It's your country."*

LDRD is a critical tool in providing "the right stuff" to attract and engage the best and brightest talent to work on national security problems.

## Performance Against DOE Objectives

The present form of LDRD was authorized by Congress in 1991 (P.L. 101-510, Section 3132) to be the sole source of discretionary research funds. It is implemented under DOE Order 413.2A, "Laboratory Directed Research and Development," which identifies the following five objectives for the program:

- maintain scientific and technical vitality of the Laboratories,
- enhance the Laboratories' ability to address future DOE missions,
- foster creativity and stimulate exploration of forefront science and technology,
- serve as a proving ground for new research, and
- support high risk, potentially high value R&D.

One measure of the FY 2005 LDRD Program is performance with respect to these objectives.

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1. B. Pimentel, "Sandia Software Could Help Border Security,: San Francisco Chronicle, p. E-1, October 31, 2005.

2. C. Hedden, What Makes for a Good Job?" *Aviation Week & Space Technology*, p. 52, May 3, 2004.

***Scientific and Technical Vitality***. LDRD is essential to attracting and retaining young staff. In the last four years, almost a quarter of Sandia staff supported LDRD projects. Close to half of the full-time employee (FTE) hours spent on FY 2005 LDRD projects are from staff with fewer than five years of experience. In addition, FY 2005 LDRD projects supported more post-doctoral appointments (67%) than any other program at Sandia, and nearly 80% of those post-docs were converted to full-time Sandia employees.

***Future DOE Missions***. LDRD is essential to creating the capabilities that bring customers to Sandia, both new and returning. For example, the LDRD Program anticipated the need for research into technologies to counter chemical or biological warfare agents and other problems in homeland security several years before the 9/11 attacks. It has become apparent that the needs for biological science and technology in support of national security include prevention, interdiction, crisis management, protection, consequence management, and attribution. In FY 2005, the growing portfolio of LDRD projects in biological science and technology exceeds $7M, including a multimillion dollar Grand Challenge project.

***Forefront Science and Technology***. In addition, LDRD projects in FY 2005 provided approximately 800 opportunities, through publications and presentations, to communicate the cutting-edge advancement of science and technology to outside institutions.

***New Research***. The importance of biotechnology to national security is now recognized, for instance, in defense against biowarfare and bioterrorism. In fewer than ten years, LDRD has placed Sandia's biotechnology initiative on the map. Sandia's biotech publications have grown from virtually nothing to rivaling the impact of the other national laboratories.

***High Risk, High Value R&D***. LDRD projects conduct high risk research that would not be funded by programmatic sources, i.e., the DOE or other federal agencies. Often, after an LDRD project reduces the initial risk, another sponsor will fund follow-on work.

## Performance Against OMB Measures

Another way to measure the LDRD Program is to compare it against the three criteria stipulated by the OMB and the President's Office of Science in 2002[1]: Relevance, Quality, and Performance.

***Relevance***. Sandia's LDRD Program is designed to fund mission-relevant R&D. Investment area teams issue calls for new R&D ideas; there is a strong programmatic component to the proposal reviews; and the investment areas often jointly fund projects of mutual interest. In addition, all Sandia LDRD projects are reviewed by the National Nuclear Security Administration Sandia Site Office (SSO) to affirm their ties to the missions of the DOE.

***Quality***. The quality of the program is represented by external recognition, publications, and intellectual property. In calendar year 2004 (the last complete year for which data are available), the Sandia LDRD Program produced 429 refereed publications. In the period from 2000 through 2004 (the most recent 5 years for which data are available), Sandia LDRD produced 564 papers, of which 70% were cited one or more times. The Institute for Scientific Information's impact metric measures the impact on the scientific community by tallying the number of times published research is cited by other researchers. Sandia's LDRD related optics and acoustics publications from 2000 through 2004 were ranked first among the 140 institutions measured. In engineering mathematics, LDRD papers were ranked third among all of the institutions measured. In both civil engineering and nuclear engineering, LDRD papers were ranked sixth among all of these institutions. Three, including the first and second of the five most highly cited Sandia papers in 2004, were from LDRD projects. In addition, LDRD projects received 8 additional awards of recognition from external institutions.

In terms of intellectual property, in FY 2005 the LDRD Program was responsible for over 40% of Sandia's total number of technical advances and over a third of Sandia's issued patents. Since 1992, the number of patents granted and copyrights issued has increased substantially. In FY 1992, Sandia submitted 15 patent applications, 49 patent disclosures, and 6 copyrights. The FY 2005 LDRD Program yielded the submission of 84 patent applications, 210 technical advances, and 18 copyrights.

***Performance***. The FY 2005 program is funded at approximately $109M, which is 5.7% of the total operating budget.[2] Approximately 81% of the projects are defense-related; 66% are identified as potentially dual-use (i.e., applying to both defense and non-defense).

1. J. Marburger and M. Daniels, "FY 2004 Interagency Research and Development Priorities." Memorandum for the Heads of Executive Departments and Agencies. Washington DC, May 30, 2002.

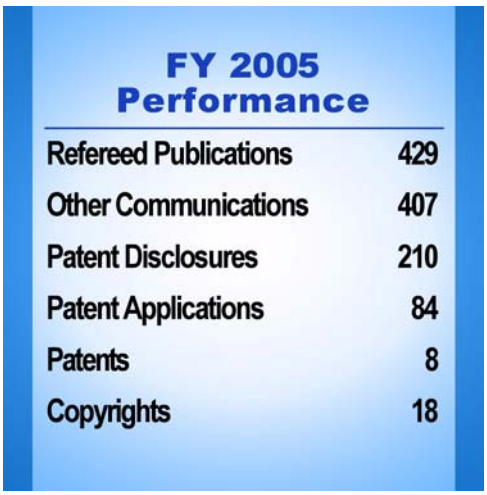
The program closed FY 2005 within 1% of its projected cost.

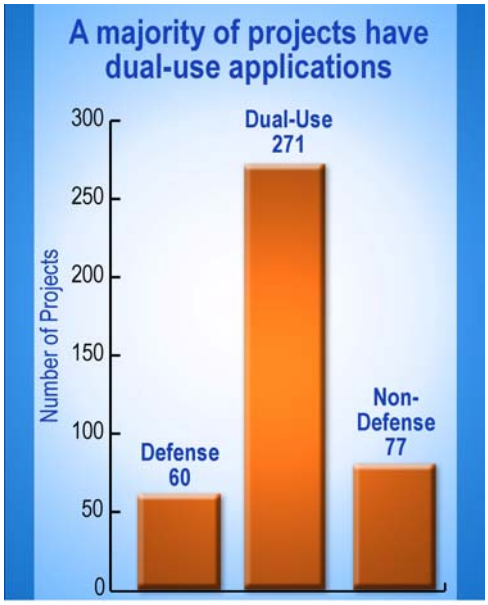
A measure of project performance is progress against identified research goals and milestones. Project milestones are determined for approximately every $300K of funding and identify specific accomplishments in research progress. Goals refer to certain capabilities that are desired within the overall framework of a project's research activities. In scientific inquiry, meeting all of the intended milestones and goals indicates that the research challenges have been set too low. So project teams are encouraged to create a deliberate mismatch between the group's research aspirations and its present knowledge and resources. In FY 2005, over 95% of LDRD projects met at least two-thirds of their milestones, and over 80% substantially met or achieved their goals. This indicates that while researchers work hard to meet projected goals and milestones, there is a still a high level of "stretch" anticipated by LDRD participants in their research activities.

## FY 2005 Program Strategy and Process

In January 2004, Sandia's Mission Council approved the organization of the FY 2005 LDRD portfolio into four categories of investment areas: Science, Technology and Engineering; Mission Technologies; Grand Challenges; and Corporate Objectives. Within these categories, the individual investment areas are:

- Science, Technology, & Engineering Investment Areas: Advanced Components and Certification Engineering, Advanced Manufacturing, Biotechnology, Computational and Information Sciences, Chemical and Earth Sciences, Electronics and Photonics, Engineering Sciences, Materials Science and Technology, Pulsed Power and High Energy Density Sciences, Science and Technology Strategic Objectives



2. DOE Order 413.2A limits LDRD research funding to not more than 6% of the Laboratories' total operating budget for the year, including non-DOE-funded work. The Order also allows funding of capital equipment for LDRD not to exceed 6% of its total capital equipment budget for the year, but Sandia's LDRD Program does not assess capital equipment. The figures provided do not include the costs of construction and capital equipment. The program is slightly less than 6% because it is planned conservatively in the previous FY.

- Mission Technology Investment Areas: Energy and Infrastructure Assurance, Homeland Security, Military Technologies and Applications, Nonproliferation and Assessments
- Grand Challenges
- Corporate Objectives Investment Areas:: Advanced Concepts, Seniors' Council, University Collaborations

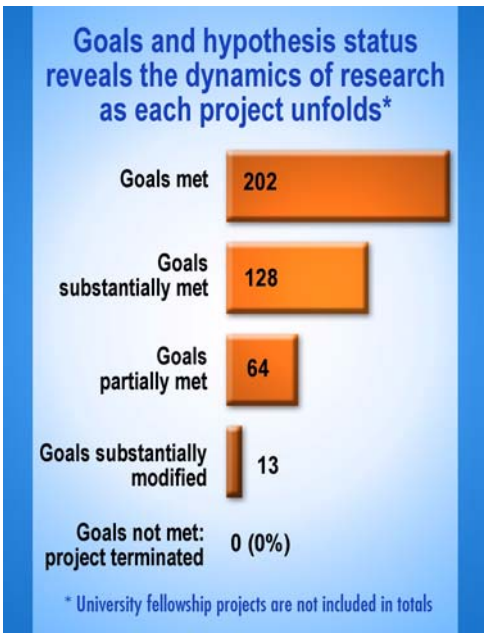
The Science, Technology, & Engineering investment areas principally support the nuclear weapons mission of the Laboratories, while the Mission Technology investment areas principally support other missions. However, the nature of most LDRD projects is such that multiple missions typically benefit from any given project.

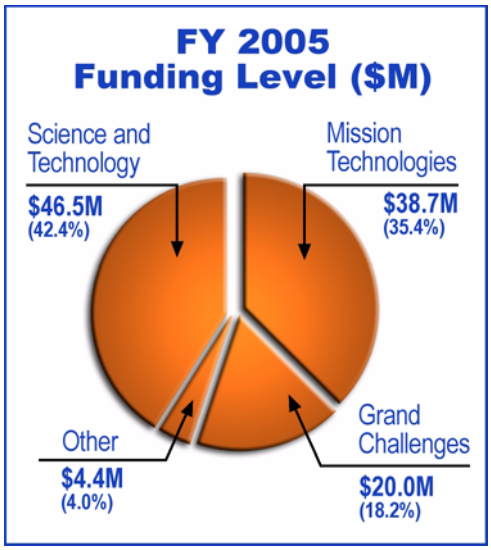
The Grand Challenges investment area is composed of large (greater than $1M) multidisciplinary projects that address the difficult and complex R&D issues that are vital to the core purpose of the Laboratories.

The Corporate Objectives investment area includes Advanced Concepts, University Collaborations, the Presidential Early Career Awards for Scientists and Engineers (PECASE), and the Seniors' Council investments. The Advanced Concepts and Seniors' Council investments support investigations related to potential future threats to national security. The University Collaborations and the Presidential Early Career Awards not only keep the LDRD Program tightly connected to cutting edge research at universities, but they also aid in maintaining the vitality of Sandia's workforce.

The process for selecting FY 2005 LDRD projects began in March 2004 with a call for proposals from each of the above investment areas. In response, 1034 brief ideas for proposals were submitted. The investment area teams reviewed these ideas and selected 442 for development as full proposals. The two-step idea-proposal format streamlines the process, encouraging more ideas to be submitted and decreasing the time spent writing and reviewing unsuccessful proposals.

The proposals are reviewed for technical and programmatic merit. Technical review criteria include scientific and technical soundness, creativity and innovation, and project planning. Programmatic review criteria may include potential customer needs, the opportunity to close gaps within the existing technology base, and prospective leveraging of other investments. From these proposals, 223 projects were selected by the investment area teams in June for funding.



FY 2005 ended with 410 active projects, 221 in their first year, 116 in their second, and 68 in their third. On average, FY 2005 projects were funded at approximately $270K, with the smallest project at $5K and the largest at $3.7M.

## Additional Reading

A comprehensive discussion of DOE and Sandia's LDRD Program management and funding sources may be found in the "Sandia National Laboratories FY 2005–2010 Institutional Plan."

**FY 2005 Selected Statistics**

| | |
|---|---|
| Full-time Staff | 431 |
| Smallest Project ($K) | 5 |
| Average Project ($K) | 270 |
| Average Project (FTE) | 1.06 |
| Largest Project ($M) | 3.7 |
| 1st Year Projects | 221 |
| 2nd Year Projects | 116 |
| 3rd Year Projects | 68 |
| 4th Year Projects | 3 |

# Sandia National Laboratories' FY 2005 Laboratory Directed Research and Development (LDRD) Program: New Directions!

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

Sandia National Laboratories has made significant contributions to America's science and engineering base since our creation in 1949. Our culture, originally defined by the oversight provided by Western Electric and then Bell Laboratories, put a premium on science with a specific focus on our weapons engineering responsibilities. Sandia's physical, engineering, and computational expertise was built to support the nuclear weapons program, but it also provides technical solutions for the mission needs of the Department of Energy (DOE) and Other Federal Agencies. Shifting national security priorities, and looming concerns about energy security, economic prosperity, and homeland defense, require that Sandia take a leadership role in the future of science and technology (S&T) to support the needs of the nation.

*"We see science and technology as a true way to help the nation move into the future."*
(Tom Hunter, President and Director of Sandia National Laboratories)

As the Labs' only discretionary research and development (R&D) program, LDRD is critical to Sandia. LDRD supports staff-generated, leading edge R&D that enables an accelerated transition of innovative technical solutions to some of our nation's most challenging problems. Through LDRD, Sandia can build and maintain its capabilities and workforce to be at the forefront of S&T. LDRD is flexible enough to be anticipatory of the nation's needs, as well as responsive to emerging threats. LDRD enables Sandia to look to the future and take risks that other R&D programs cannot. LDRD allows Sandia to build critical S&T capabilities and to define the scope of potential applications. The impact of the LDRD Program is often demonstrated through scientific advances and technological innovations based on fundamental new understanding, where the impact reaches far beyond the originally intended purpose. LDRD projects initiated years ago are having multiple and sustained benefits for DOE, Department of Homeland Security, and Work for Others customers.

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

LDRD is addressing critical Stockpile Stewardship R&D issues ranging from materials to microelectronics to accelerated aging and stress tests of silicon microsystems. These have the potential for enormous impact on the National Nuclear Security Administration's mission to maintain and enhance the safety, security, reliability, and performance of the US nuclear weapons stockpile. These same LDRD projects will likely impact other missions because of the ubiquitous need for improved packaged microsystems in many government, military, and commercial applications. Similarly, the development of microelectromechanical devices, ranging from complete gas or liquid phase chembio sensors to highly sensitive mass spectrometers, has the potential for applications in nonproliferation and homeland defense, as well as for innovative ion traps for quantum computing.

There are many other examples of LDRD projects that have the potential for enormous impact to national security; however, the demand for technical solutions frequently occurs at a faster pace than can be anticipated or achieved. To promote technological innovations in alignment with Sandia's strategic missions, we must seek improvements in the LDRD Program.

After consultation with Sandia's Executive Office and a workshop with LDRD participants and stakeholders, it was decided that it was time to seek fundamental changes in LDRD, including a renewed em-

phasis on differentiating science, risk-taking, and innovation in alignment with Sandia's strategic missions. Our high level strategy for LDRD in FY 2007 consists of three pillars

(1) To promote differentiating science and risk-taking R&D in alignment with Sandia's strategic missions

(2) To nurture and grow our S&T base to sow the seeds for the future

(3) To ensure our LDRD investments are aligned with our corporate strategy

Additional changes to LDRD include reducing the number of investment areas to improve process efficiency and integration, and increasing the focus on managing project outcomes. These improvements should result in higher risk and better quality LDRD projects that will allow Sandia to maintain its S&T leadership role that will truly support the needs of the nation.

*"...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."*

# Science, Technology, and Engineering

The LDRD program helps Sandia maintain its position as a world leader in science, technology, and engineering (ST&E). The objectives of LDRD's ST&E investments are 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. The ST&E investment areas 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 in FY 2005 include the following:

- 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 Information Sciences
- ST&E Strategic Objectives
- Biotechnology

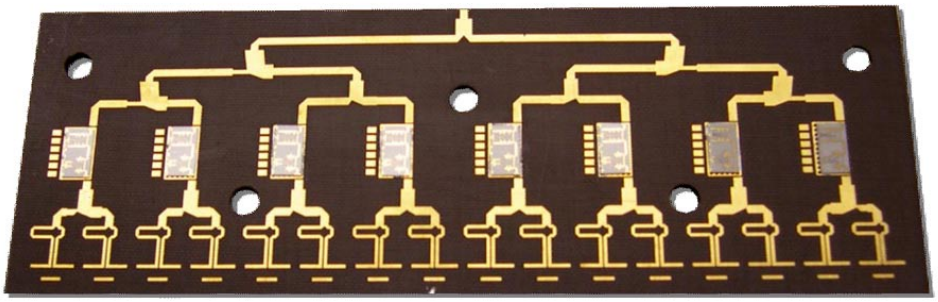
ST&E investments in **Materials Science and Technology** work to develop innovative materials and materials processing concepts to enhance Sandia's ability to steward the nuclear weapon stockpile and to minimize or mitigate possible national security threats. Projects also focus on understanding predictive performance, aging, and reliability of new and existing products and components.

Pursuits in **Advanced Manufacturing** specifically support the advancement 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 ventures in **Pulsed Power and High Energy Density Science** that focus on innovative ideas that 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. Specific applications and missions include inertial confinement fusion, hostile nuclear environment survivability, electromagnetic propulsion and launching, defeat of biological agents, and inertial fusion energy.

Investments in ST&E are also encouraged to perform multidisciplinary work that builds on our differentiating strengths to achieve integrated processes and technologies. For example, project



**67028 "Passive Electronically Steerable Array (PESA) for Miniature Synthetic Aperture Radar (miniSAR), Precision Guidance, and Intelligence/Surveillance/Reconnaissance"**

Researchers assembled an electronically steerable array (ESA) prototype consisting of 16 quasi-yagi antenna elements with 8 MEMS phase-shifters that enable compact size, low loss, and increased functionality.

**67028 "Passive Electronically Steerable Array (PESA) for Miniature Synthetic Aperture Radar (miniSAR), Precision Guidance, and Intelligence/ Surveillance/Reconnaissance (ISR)"** combines advancements in advanced component manufacturing and engineering, electronics, and military technologies.

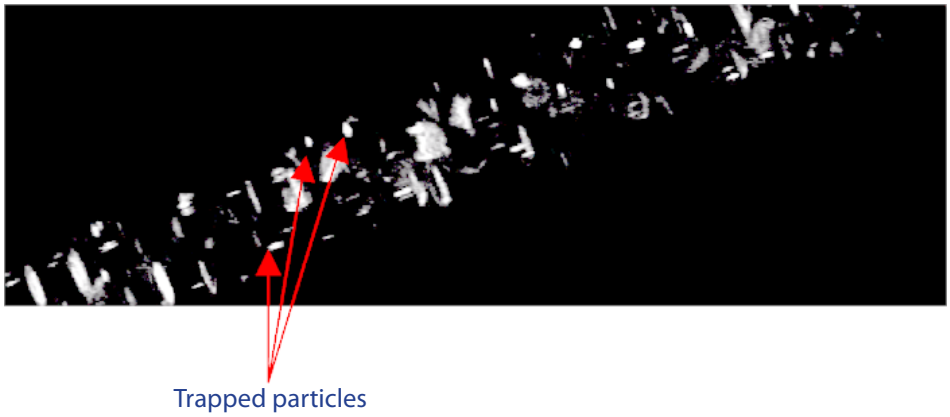
The project aims to develop an antenna system for miniSAR systems and to facilitate radar use for precision guidance, tactical unmanned aerial vehicles, and other intelligence/surveillance/reconnaissance applications. Researchers are working to accomplish this by using a passive array and inserting a microelectromechanical system (MEMS) phase shifter into the antenna feed path to create a compact, low loss, lightweight PESA. Development and assembly of the antenna demand advancements in fabrication, packaging, electronics, engineering, and integration. Progress in technology development and integration includes improvements to the MEMS components and modifications to the 16 element array to produce cross-polarization isolation, sidelobe levels, main beam width, and main beam peak that meet or exceed specification for a SAR imaging system. Researchers assembled and tested performance of a one-dimensional PESA, assembled a two-dimensional modular card antenna array system, and demonstrated a 4-bit phase shifter. The research and development for the PESA will impact technology at the component, device, integration, and system levels.

Other investments in **Electronics and Photonics** focus on sensor concepts that advance semiconductor science and technology and seek to provide the capability to eventually realize small, low power, highly integrated microsystems for national security applications.

**Engineering Sciences** research works to advance 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. Recent focus in engineering sciences attempts to explore micro- and nanoscale phenomenology definition and application, including advanced microsystem engineering processes.

The 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, MEMS, biotechnology, and homeland security. Some investments in this area are recently focusing on informatics related to graph theory, biological coding theory, pattern recognition, image and language processing, engineered collectives (virtual and robotic), and symbolic computation. Additionally, recent advances in massively parallel algorithms and architectures are already greatly expanding the complexity and usefulness of simulations.

Another key strategic goal of the 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. For example project **79889 "Nanodielectrokinetic Chromatography (nanoDEKC): Rapid Separation and Classification for Biodefense and Biomedical Applications"** works to create the potential for continuous,




**79889 "Nanodielectrokinetic Chromatography (nanoDEKC): Rapid Separation and Classification for Biodefense and Biomedical Applications"**

When the voltage was dropped to zero, the trapped particles were released in a concentrated plug.

autonomous and low false-positive operation in a sensor. Achieving this technology would provide the capability for a detect-to-warn system that would represent an important contribution to biodefense programs. The technology would also be able to contribute to non-proliferation efforts including monitoring effluents of suspect facilities, and could also be used to monitor water supplies. Thus far, researchers demonstrated the feasibility of a new system for rapid concentration and separation of vegetative cells, spores, viruses, and other particulates. They optimized the fabrication of a simple, cost effective, and extremely versatile system that allows exploration of new areas such as nanodielectrophoresis (nanoDEP) and nanoDEKC. In FY 2005, researchers studied the key parameters responsible for microparticle manipulation in the device and analyzed non-conventional materials to determine suitability for nanoDEP.

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.

# Advanced Components and Certification Engineering

## Predictive Accelerated Aging of Microsystems: The Science of Dormancy

*52727*

*M. T. Dugger, R. A. Plass, J. V. Cox, D. M. Tanner, M. A. Duesterhaus, B. Jokiel Jr., T. J. Skousen, K. A. Klody, J. A. Ohlhausen*

### Abstract

Qualification of microsystems for defense applications is critically dependent on our ability to build confidence in their performance by accurately predicting the evolution of their behavior over time in the stockpile. In this project, packaged surface micromachined devices are subjected to accelerated aging to explore dormancy and operational environment effects on reliability and performance. We can accelerate failures in complex microelectromechanical systems (MEMS) devices using elevated temperature and humidity at temperatures below those required to cause monolayer decomposition. Failure of rotating gears appears to be associated with an adhesion site in the hub where the nominal gap is 0.3 µm. The apparent adhesion energy changed from 10–17 $\mu J/m^2$ for as-coated samples to about 5 $mJ/m^2$ for aged samples. We found surface monolayer coverage to be reduced on hidden areas compared to exposed areas. This suggests that saturation coverage may be difficult to achieve in areas where some tribological contacts in devices take place. Adhesion and friction behavior, determined using a surface micromachined diagnostic structure, showed that friction force and adhesion were proportional to monolayer coverage in the range examined. We used finite element modeling to estimate the contact areas and adhesion forces that would occur between a gear and hub. We incorporated the resulting estimates of adhesion forces into rigid body dynamics models of a gear on a hub. For the highest simulated adhesion and friction coefficients, the actuator had ample power to drive the gear, suggesting that adhesion between the gear and hub alone is not responsible for the failure of devices observed after aging. Integration of the aging data and the modeling approach for surface micromachined devices provides a toolset that we can apply to predict the behavior of packaged microsystems after storage.

### Accomplishments

We packaged approximately 1100 surface micromachined polycrystalline silicon die for this project, representing the largest volume of packaging of a single device for any project thus far. We investigated low temperature glass lids for creating a hermetic environment with the capability for visual inspection, but this process could not be made reliably hermetic. Although not hermetic, functional testing showed that the low temperature lid sealing process eliminated the yield loss normally experienced in packaging. This result validates that high temperature lid seal processes are detrimental to device yield, and that low temperature lid sealing methods should be developed to improve reliability and yield.

We used time-of-flight secondary ion mass spectrometry (ToF-SIMS) to quantify the coverage of monolayers used to mitigate in-process adhesion, since it has the spatial and structural specificity needed to perform analysis at the device level. Although it was previously assumed that monolayer saturation is achieved when ellipsometry no longer shows a change in thickness, ellipsometry measurements are made on

single crystals to avoid roughness effects and on surfaces directly exposed to the molecules during deposition. Results of ToF-SIMS analysis of polysilicon flaps treated with vapor-phase tridecafluorotri(dimethylamino)silane (FOTAS), and liquid-deposited perfluorodecyltrichlorosilane (PFTS) showed the following: (1) PFTS coverage is reduced in hidden areas compared to exposed areas, and (2) the FOTAS molecule fragments differently on hidden areas compared to exposed areas, possibly due to differences in coverage or roughness.

We exposed FOTAS coated devices to temperatures ranging from 200 to 250 °C at 500 ppm water vapor, and for durations between 500 and 2000 hours. ToF-SIMS showed that this exposure caused a reduction in monolayer coverage. The adhesion and friction behavior, determined using a surface micromachined diagnostic structure, showed that friction force and adhesion were proportional to FOTAS coverage in the range examined. We expect that the interface behavior will stabilize at higher coverage. These results indicate that operational or storage conditions that cause a small change in monolayer coverage can lead to a large increase in friction and adhesion. The data suggest a 74% increase in coefficient of friction and an 80% increase in adhesion force for a 15% decrease in monolayer concentration.

We used functional tests of various actuators and MEMS devices to evaluate the effects of aging on MEMS structures. We can accelerate failures in complex MEMS devices using elevated temperature and humidity at temperatures below those required to cause monolayer decomposition. The trend indicates longer life at lower temperatures and humidity levels. Failures of the torsional ratcheting actuators, or TRAs, are due to an adhesion site in the hub of the load gear where the typical gap is 0.3 µm. We observed a change in the apparent adhesion energy for aged devices, measured using micromachined cantilever beams. The apparent adhesion energy changed from 10–17 $\mu J/m^2$ for as-deposited FOTAS to ~5 $mJ/m^2$ for aged samples. There was significant variability in adhesion from package to package, with some exhibiting no change with aging. We found these to contain small particles on the contacting surfaces that reduced the real contact area and minimized adhesion.

We used finite element modeling to estimate the contact areas and adhesion forces that would occur between a gear and the vertical section of a hub under different conditions. We incorporated the finite element estimates of adhesion forces into rigid body dynamics models of a gear on a hub, created using LMS Virtual Lab software. We allowed the modeled gear, supported by mechanical contact definitions, to wobble in accord with the tolerances created by device fabrication. We incorporated adhesion (determined experimentally for various environments) with a user subroutine. For the highest adhesion and friction coefficients considered, the TRA had ample power to drive the gear. These results indicate that adhesion at the gear-hub interface alone is not responsible for the failure of devices observed after aging.

## Refereed

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## Surety and Accountability Enhancements for Storage Containers

*52728*

*J. W. O'Connor, S. C. Lindblom, K. J. Hearrean, J. R. Greegor, S. K. Namperumal, T. E. Bennett, M. C. Jackson, C. L. Turner, R. J. Punnoose*

This is a classified report.

# Nuclear Safety Weaklinks for Thermal and Mechanical Environments

*52729*

*A. C. Hall, S. J. Glass, G. R. Lyons, K. Rahimian, K. Eras, R. Tandon*

## Abstract

Using a nuclear safety theme that employs first principle, positive measures, Sandia adheres to four nuclear safety design principles: incompatibility, inoperability, isolation, and independence. A weaklink is an element vital to the detonation process that is rendered irreversibly "inoperable" by an abnormal environment. The objective of this project was to develop new materials and technologies as weaklinks for abnormal thermal and mechanical (shock or crush) environments.

We pursued a thermal weaklink based on a new dielectric material that converts to a conductor at elevated temperatures. The resistivity of this material changes by over five orders of magnitude in response to a thermal insult; therefore, it could be used to short a capacitor. We identified and characterized the best formulation. We developed a technique for making thick films of the material on various substrates and optimized it for glass. We confirmed and optimized film quality for thick films. We studied the potential for using these materials as the insulating layer for flat cables. Attempts to build a demonstration weaklink were unsuccessful due to materials incompatibilities.

We also studied and developed a shock/crush weaklink based on engineered stress profile (ESP) glass. This material fractures predictably at a specific stress level. It can be metallized, allowing it to act as part of a circuit that would be destroyed at a critical shock or crush load. We investigated implementation of the glass as part of the critical circuit in the stronglink. We fabricated two geometries/designs and tested one in shock loading. We also investigated implementation of the glass as a frangible element in a capacitor. In this concept, the mechanically induced fracture of the glass element in the capacitor short circuits it. We identified and tested new glass compositions for their strength and fragmentation properties after ion exchange.

## Accomplishments

- Thermal Weaklink

We identified and characterized the best formulation based on silver nitrate/polyvinyl alcohol. Initial studies of coated glass substrates for use with flat cables suggested that film thickness was insufficient. We optimized thick film deposition of the material and identified the maximum film thickness. We developed coated substrates with several micron thick films. We made attempts to use these materials as insulators for flat cables, but they proved to be incompatible with copper metal, oxidizing copper on contact and reducing the silver nitrate to silver metal.

We studied other metals for making a thermal weaklink based on the concept of metal ion reduction by polyvinyl alcohol. Copper was the best candidate, and we studied several copper salts. Copper films were made using this methodology, but film thickness was insufficient. We studied various methods for increasing film thickness, but none were successful.

- Mechanical Weaklink

We concluded additional processing studies on the Corning 0317 glass composition. These included ion exchanges at a higher temperature (550 °C) for several time durations. Strength, fragmentation, central tension, and diffusion depth results were compiled for all processing conditions studied. We measured and compiled strength variability under different fracture conditions for thermally tempered glass with different thicknesses and degrees of thermal temper. We explored nanoindentation measurements for determining surface stresses. We investigated fragment size distributions and patterns using fractal analyses and simulations of fragmentation using measurements of crack branching angle and distance and number of crack branches. We compared fractal analyses of fragmentation for both punch induced and biaxial strength test fractures.

We investigated alternative glass compositions. We conducted sodium for lithium ion exchanges for the SCHOTT ROBAX® glass composition as a function of glass thicknesses, and ion exchange time and temperature. We characterized central tension, ion exchange depths, and strength and fragmentation behavior. This glass gives good strengths; however, it requires longer exchanges to achieve the same level of central tension and degree of fragmentation obtained for 0317 glass.

We examined several processes for making samples with asymmetrical stress profiles or making selected regions of the glass weak relative to an ion exchanged surface. These included coating regions of the glass with a material that prevents ion exchange, removing some of the surface after the ion exchange, and bonding small pieces of glass to larger sections and exchanging them together. Coating materials included indium tin oxide (ITO), $Al_2O_3$, TiN, SiN, and Pt. The coating thicknesses were in the range of 1000 Å and were sputtered or evaporated onto the surface. The ITO and Pt coatings had a minimal effect on blocking ion exchange, or they came off during the exchange. The $Al_2O_3$ and TiN coatings both significantly blocked ion exchange and shifted the stress profiles as intended.

We investigated processes for making parts with more complex geometries, including bonding one piece of glass to another using traditional glass blowing techniques and a new low temperature glass bonding process developed by SCHOTT. Low temperature bonding was done either prior to or after ion exchange.

We evaluated three approaches for implementing stressed glass as a mechanical weaklink or shock enhancer. Two were based on using stressed glass as a frangible substrate for critical electrical leads in the stronglink. We selected the glass geometry, thickness, and orientation to produce glass fracture and destruction of the electrical leads at a critical mechanical shock load. In the third approach, we incorporated a thin glass strip in the windings of a capacitor. Deliberate fracture of the glass led to its fragmentation, damaging the capacitor materials and inducing a short circuit.

## Refereed

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# Novel and Robust Environmental Sensing Devices (ESDs)

*52732*

*J. A. Mitchell, N. S. Finch, J. W. Franklin, J. W. Wittwer, D. C. Graf, R. C. Clemens, R. A. Renn*

## Abstract

In this research project we designed, fabricated, and tested novel inertial environmental sensing device (ESD) prototypes using microelectromechanical systems (MEMS) technologies. The primary focus was on the development of novel designs, fabrication, packaging, and testing of ESDs targeting low-g (< 20 g) applications using LIGA (for the German term *Lithographie, Galvanformung, und Abformung*, for lithography electroforming, and molding) and wire electrical discharge machining (EDM). We investigated bistable compliant mechanisms and eddy current damping for use in inertial sensing devices.

## Accomplishments

This year the team focused on damping and compliant mechanisms in inertial sensing devices. Modeling and simulation was a critical aspect in the development of designs.

Damping vibrations is an important element in some types of inertial sensing devices and can be implemented using magnetic forces generated by a static magnetic field interacting with eddy currents. We developed a finite element model for the analysis of eddy currents in a thin sheet of conducting material. We used this model for design and sensitivity analyses. We fabricated a demonstration prototype consisting of a shuttle mass and tethering springs to validate the model. We measured damping levels in the prototype using laser Doppler velocimetry (LDV) and found them to be above critical as predicted by the numerical model.

The second area of focus was the design, analysis, fabrication, and testing of novel compliant mechanisms fabricated using LIGA and wire-EDM. We fabricated and tested several novel monolithic topologies. Using finite element modeling and measured data, we conducted uncertainty and model validation studies on the "X-Wing," "Pi-Wing" and "B-Wing." These topologies utilize carefully selected geometries to create almost bistable mechanisms that are useful in inertial sensing devices. This spring softening effect enhances the switch performance by lowering the contact resistance through higher contact forces.

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# Advanced Neutron Monitors for JTA and Stockpile Monitoring

*52733*

*N. Mascarenhas, D. A. Sunnarborg, J. Peel, W. Mengesha*

## Abstract

Current joint test assembly (JTA) neutron monitors rely on knock-on proton type detectors that are susceptible to x-rays and low energy gamma rays. We investigated two novel plastic scintillating fiber directional neutron detector prototypes. One prototype used a fiber width less than 2.1 mm, which is the range of a proton in the fiber. We used the asymmetry in the recoil proton energy deposited in the fiber to determine the incident neutron direction. The second prototype measured both the recoil proton energy and direction. We determined the neutron direction from the kinematics of single neutron-proton scatters.

## Accomplishments

We studied two types of directional neutron detectors using plastic scintillation fibers. The fibers used were Saint-Gobain BCF-12 fibers. We used a VNIIA 14 MeV pulsed D-T neutron generator in this study. The neutron generator pulsed at 100 Hz with a pulse width of 100 microseconds. The neutron flux was $8.2 \times 10^6$ neutrons/s. The first detector was an energy spectra detector that had a poorer angular resolution (about 45 degrees). We studied various fiber shapes: round and square cross sections and 0.5 mm and 1 mm sizes. We used a "t" test to compare the detector performance. The detector showed optimal performance with 0.5 mm round fibers.

The second detector was a novel tracking neutron detector that used 64 0.5 mm x 0.5 mm x 100 mm square fibers. It was sensitive to changes in neutron source direction of 10 degrees. We studied the directional performance in the presence of x-rays. We used a Kevex x-ray generator to simulate x-ray background present during JTA tests. We used x-rays with a 90 KeV end point spectrum. The tracking detector showed good directional performance in an x-ray to neutron background of 1,000,000:1.

We simulated the tracking detector using GEANT4. The simulations suggested using two fiber hits with > 5 MeV in one fiber to reconstruct proton recoil tracks. We computed the intrinsic detection efficiency for these events to be 0.374%.

Due to limitations in readout electronics, we could only capture one neutron scatter event per pulse from the neutron generator. This readout limitation could be overcome in the future by using a flash analog-to-digital converter and ring buffer to digitize each channel. This could enable the use of such a detector in a high neutron rate environment. To summarize, we demonstrated a directional 14 MeV neutron detector that is compact, has good angular resolution, and is immune to x-ray background.

## Refereed

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# MEMS/Fuze Diagnostic Extraction in High-g Environments

*67118*

*A. Mittas, E. A. Henry, R. R. Lockhart, T. L. Martinez*

## Abstract

Recent world events and military needs have given rise to durable earth penetrating weapons. In order to characterize the high load that components experience in earth penetration events, one needs the capability to: (1) record the high deceleration event for post test analysis, (2) actuate or control components during the deceleration event, and (3) record the functioning of the experiment during the penetration event. The microelectromechanical system capacitive discharge unit (MEMS/CDU) Diagnostic Extraction System (MDES) is designed to perform these three functions in high deceleration environments.

## Accomplishments

During the two year project, the MDES data recorder went through two design iterations in order to fit in the intended volume. The second and final iteration incorporated new technology and circuit design never before used in high g penetrator data recorders. Some of the new technology included 3.3 V digital circuits that interfaced with 5.0 V analog circuits using level shifting devices. We included high voltage (10 V) digital inputs and digital outputs to control and actuate experiments during penetration events in the design. Our design included more versatile adjustable gain and adjustable offset on analog amplifiers that would allow us to install new accelerometers on the external accelerometer block when they are destroyed in testing. We also included four analog channels for the experiment so that the recorder could record experiment functions during a penetration event. When the recorder is not used in conjunction with an experiment, we can use the experiment channels as accelerometer or strain gauge channels offering the experimenter six channels of acceleration to record. The most significant design accomplishment for the MDES data recorder was the use of flex circuit interconnects to interface the three boards of the data recorder together. We fabricated the flex interconnects along with the printed circuit boards so that the entire data recorder is one interconnected system, as opposed to three separate printed circuit boards that require some other interconnection method.

We qualified the data recorder using a drop table. The recorder interfaced to two mechanical non-volatile memory (MNVM) devices and two SiRES (silicon reentry sensor) g switches. The recorder applied power to the experiment prior to the drop and successfully recorded the experiment function during the 2.0 kg drop deceleration. Both the experiment and recorder survived the qualification event.

We tested the MDES data recorder and MNVM experiment together at the Waterways Experimentation Station (WES). The test consisted of the data recorder and experiment riding an approximately four inch diameter subscale penetrator. The data recorder and experiment were powered separately; however, the recorder turned on power to the experiment. We fired the penetrator into a 3.4 ksi concrete target at 855 ft/sec. The resulting penetration in the target produced a peak g level of approximately 7.19 kg and an average or mean level of approximately 5.7 kg with a duration of approximately 5 ms. Both the data recorder and experiment functioned during the launch and penetration environments, and both survived the test.

During the test, the recorder recorded x (axial), y, and z (lateral) loads on the penetrator, the capattery power of the data recorder, the trigger location in the record time window, the functioning output of the MNVM, the static MNVM contents, and the actuation of the SiRES g switches.

# Next Generation High Voltage Switches for Capacitive Discharge Firing Systems

*79868*

*A. W. Roesler, K. Bunch, B. Wroblewski, C. A. Walker, T. A. Friedmann*

## Abstract

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 focuses 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 create 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. Initially, we used silicon as a switch substrate to provide the greatest flexibility with current processing methods. We found conventional high temperature brazing methods to be incompatible with silicon to create a vacuum seal required for proper switch operation. A move to a ceramic substrate solved the brazing issues while maintaining compatibility with microfabrication techniques.

The ceramic substrates used are available commercially and are relatively inexpensive. Plated through-holes provide connections to the active switch elements that in current systems are built by hand. Several designs using deposited graphitic carbon passed tests for hermiticity (vacuum compatibility) and high temperature brazing. These designs are comparable to current switch designs and are intended to match their operation while demonstrating key elements of the project. We also fabricated further designs using thin film diamond films. Their operation is based on the emission and breakdown properties of diamond to initiate switching. Diamond is intended to improve the switch lifetime of graphitic films currently used as active trigger elements.

We are integrating an ultrahigh vacuum chamber system as well as a computer-controlled data acquisition system to collect data on the switch designs fabricated and to verify switch operation. The vacuum system is intended to collect data without the need for vacuum packaging so that the turnaround time on testing novel switch concepts is reduced.

## Accomplishments

- *Milestone 1. Develop Process-Compatible Design:* We found that alumina ceramic substrates are compatible with conventional vacuum processing techniques and suitable for thin film deposition. These substrates are commercially available and currently used in applications requiring via/plane interconnects such as microwave integrated circuits. Sintered ceramic substrates are laser drilled and filled with tungsten-copper to create vias. Via placement can be held to accuracies of +/- 25 micrometers over the entire substrate area, making precise interconnect placement possible. Alumina is commonly used in vacuum compatible systems; further, it is compatible with high temperature brazing. We created hermetic seals using switch designs involving deposited graphitic carbon. The metal vias demonstrated the ability to survive high temperature brazing and maintain a hermetic seal compatible with ultrahigh vacuum. Further, the thin film graphite did not peel and degrade under brazing. Initially, we chose silicon as a substrate material because of the extensive processing capabilities developed for semiconductors. We found thin film deposition of niobium for use as electrical contacts to be incompatible under high temperature brazing. Adhesion to silicon was compromised under high temperature. We then chose alumina as a substrate material because of its compatibility with vacuum processes and microfabrication technologies. Key processing technologies compatible with this application include precise, laser drilled vias for hermetic electrical connections; vapor-phase deposition of thin films including graphitic carbon and diamond; and masking and etching used in semiconductor manufacturing. The processing technologies identified allow for precise placement of films and interconnects. Film thicknesses and composition can be precisely controlled, as well.
- *Milestone 2. Model and Design:* We modeled and designed several configurations. We created two-

dimensional electrostatic models to design packaging dimensions and electrode spacing to ensure unwanted switch breakdown under high voltage. We also created conduction models to determine current densities within the graphitic films used for switch triggering. We used these models to determine several film thicknesses and geometries used for device fabrication. We are in the process of packaging several designs based on graphitic films into testable switches. We created and deposited further designs based on the emission and breakdown properties of thin film diamond.

- *Milestone 3. Build Designs:* We completed several iterations of design/fabrication/testing. Initial designs were single shot and based on silicon substrates. We created a Schottky barrier using a niobium/doped silicon junction that vaporized upon receiving a triggering current. We fabricated and tested prototype devices for triggering without vacuum; we observed successful vaporization of the metal/silicon junction. High temperature brazing of these prototypes resulted in damaged and peeled films. Subsequent designs targeted alumina substrates because of the compatibility of alumina with conventional brazing techniques. We created several trigger designs including thin film graphitic carbon and graphite. We completed the packaging design and ordered parts.
- *Milestone 4. Evaluate Performance and Capability:* This milestone includes building test fixtures and test electronics. To this end, we acquired a data acquisition test system, with a National Instruments PXI controller and several acquisition and control boards. This system is a real time, stand-alone controller driven by Labview software. We designed a capacitive discharge firing set and ordered parts for construction. The firing set is controlled from the test system so that firing and characterization data can be automatically acquired. Further, we designed and built a custom high vacuum system for switch testing in a vacuum. Such testing will allow switch designs to be tested without having to be packaged. Quick evaluation of current and subsequent designs will be possible with the current test system.

# An Algorithm for Target Characterization

*79869*

*A. W. Roesler, T. G. Carne, C. R. Dohrmann, D. W. Kelton, M. S. Allen*

## Abstract

This project is developing an algorithm that 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. Acceleration records of penetration events can be used to characterize targets. The challenge for target characterization in applications is separation of the desired rigid body acceleration, or deceleration, from the confusing vibratory oscillations created within the weapon. The proposed approach is to cancel the vibratory motion with a sum of weighted accelerations technique (SWAT). SWAT uses a small number of sensors along weapon axes to decompose the measured accelerations into the vibratory and rigid body motion. A weighting function, developed in a laboratory testing, is designed to cancel the vibratory content. The resulting sensors and algorithm will provide real time acceleration with greater accuracy than previous approaches using frequency based filtering.

## Accomplishments

The first year objective was to show the feasibility of real time, accurate reconstruction of the deceleration of a penetrator's center of mass, using a scaled penetrator and laboratory testing; this included an evaluation of the number of necessary sensors, the sensor types, and the best locations for the sensors on the penetrator. The reconstruction of the deceleration of the center of mass would use response signals from accelerometers and strain gauges and be based on the SWAT technique. SWAT is capable of removing the confusing vibratory accelerations from the response data, leaving only the underlying acceleration of the center of mass, without using frequency filtering. We used a generic penetrator as our demonstration hardware to assess the capabilities of SWAT. Using a short impulsive force as input, we reconstructed the acceleration of a penetrator center of mass using SWAT, retaining the high frequency content of the input. By applying measured force inputs to the penetrator and measuring the response signals from the accelerometers and strain gauges, we evaluated and refined the SWAT algorithm using laboratory based testing. The highly vibratory response observed from the sensors is not untypical of actual penetration events, although it is much lower in amplitude due to laboratory testing. We compared the SWAT reconstructed acceleration with the directly measured acceleration of the centers of mass. The reconstructed acceleration compares favorably with the measured data. There is some vibratory noise on the reconstructed signal following the transient input, but the SWAT reconstruction follows the true acceleration well during the initial input, reproducing the rapid rise and fall times and the amplitude of the input acceleration.

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

*79870*

*P. J. Cole, J. L. Lenhart, D. E. Beutler, R. V. Baron*

## Abstract

Gel-Mylar capacitors are an important technology for the nuclear weapons stockpile. A critical issue facing the nuclear weapon organizations is the lack of radiation-hardened polymeric films and an understanding of the doping methodology to produce new films for future life extension programs (LEPs) and weapons systems. In addition to these components being important to nuclear weapons, the science associated with radiation-hardened materials is applicable to other Sandia (and external) technologies. Two issues are directly relevant: radiation induced conductivity, and the physics of doping and interface transport. For example, controlling radiation induced conductivity through specially designed dopants or conformal coatings could dramatically impact silicon based devices in satellite applications. Similarly, understanding dopant transport could provide a radiation-hardening or, conversely, a decontamination methodology. This project will establish the scientific foundation necessary to address radiation induced conductivity and the physics of doping.

This year, we focused on three areas of research. First, we corrected a series of misperceptions in the scientific community regarding the doping of polymeric films. Second, we established and used the necessary research techniques to perform preliminary analyses. Third, we identified and partially evaluated a series of processing strategies to yield radiation-hardened polymeric films. The results suggest that a greater focus on understanding the science behind the doping process and radiation induced conductivity is required to expand the applicability of this research to the broadest possible series of applications.

## Accomplishments

We achieved significant accomplishments in three research areas. First, we corrected a series of myths within the scientific community. This misinformation included the inability to reproduce radiation-hardened doped Mylar films on a laboratory scale, the relevance of the solvent solubility parameter to the doping process, and that the doping solvent should be chosen based on the film material. Specifically, we demonstrated that it is possible to dope Mylar with 2,4,7-trinitrofluorenone (TNF) on a laboratory scale, that there is no significant importance of the solvent solubility parameter in the doping process, and that the doping solvent should be chosen based on the dopant rather than the film material. Second, we established a series of research techniques, including a spectroscopic technique to determine dopant concentration and a cobalt-60 source radiation induced conductivity (RIC) measurement to understand the link between dopant concentration, concentration profiles, and the resulting radiation hardness of polymeric films. Third, we established simple processing strategies to yield radiation-hardened polymeric films. The results suggest that dopant transport from a carrier to a polymer film is not based solely on dopant concentration, but also on the dopant concentration in a film surface boundary layer. Based on these accomplishments and results, the broader issues of radiation induced conductivity and dopant transport will be our focus in future research.

# Micro- and Mesoscale Detonics of Explosives

*79871*

*R. J. Pahl, J. A. Palmer, E. J. Welle, A. S. Tappan*

## Abstract

There is currently a desire to implement miniaturized weapon components due to the expected advantages associated with their reduced mass and volume. For example, some devices currently under consideration for hard and deeply buried target defeat applications require explosive train dimensions ranging from hundreds of microns to millimeters. These dimensions are significantly smaller than current working weapon component designs. Currently, understandings of the limiting failure diameters, shock-to-detonation (SDT), and deflagration-to-detonation transition (DDT) distances are sufficient for the design of explosive trains with characteristic length scales of several millimeters or more. However, the data characterizing primary and secondary explosives at the submillimeter to millimeter length scale now being approached in component design are mostly qualitative in nature or have not been explored at all. Consequently, it is critically important to achieve greater understanding of explosive behavior at these dimensions; otherwise, reliable component design is impossible. It is the goal of this study to investigate and quantify the performance of selected primary and secondary explosives as a function of explosive column lengths, diameters, and initiation method. This project will use diagnostics such as streak camera photography, traditional and streak VISAR (velocity interferometry system for any reflector), and line ORVIS (optically-recorded velocity interferometry system) to measure DDT and SDT lengths and explosive output at these small dimensions. Although each of these diagnostics has been proven, their use on the length scales under consideration here presents new challenges. The results of this study will provide critical component design information as well as help to increase our fundamental understanding of explosive behavior on the micro and meso length scales.

## Accomplishments

We met all of our FY 2005 milestones and also acquired some preliminary data on the performance of some materials on submillimeter scales. We focused on experimental setup, including diagnostic equipment modifications, test fixture fabrication, charge loading technologies development, and preliminary testing. We developed diagnostics capable of resolving various explosive phenomena at the small length scales needed for this work. This includes building optic trains for camera systems having high magnification and high light throughput, adapting VISAR systems to measure expected velocities, fabrication of a custom testing chamber, and writing software for streak camera image analysis. To achieve our second goal of a working test fixture, we iterated several times through design and manufacture of two different test fixtures. The first fixture enables the evaluation of interactions between different ignition sources and energetic materials. The second fixture enables a well characterized thermal source to be applied to materials to explore the fundamentals of charge initiation and burn front propagation.

We also produced a working charge holder that is adaptable to current and future loading technologies. In our charge loading work, we investigated the new technology of inkjet printing of explosives. This technique allows very small charge columns (< 400 micron) to be filled repeatedly and reliably, which is needed for reliable test data. In our most recent work, we successfully printed two different primary explosives in 400 micron columns. Preliminary analysis of streak camera data recorded from this work resulted in the first estimates of the detonation velocity and ignition characteristics of inkjet loaded charges. We also established femtosecond laser machining as a viable method of making free-standing columns with energetic material diameters as small as 225 microns. Larger diameter columns are necessary to evaluate the performance characteristics of secondary explosives that have small detonation failure length scales. We successfully machined free-standing hexanitrostilbene (HNS) columns that will be tested to measure HNS material properties at these scales. Finally, the output characteristics of materials reacting on larger length scales must be known to determine the effects of reducing charge size. For most materials, these parameters are known and are found in the literature. However, for some materials such as the primary explosives investigated in this work, these properties are unknown. We conducted VISAR testing to quantify the output of several primary explosives, thus providing baseline data for future comparison.

# Embedded Neutron Generator Health Microsensor

*79872*

*D. S. Epp, R. A. Damerow, S. T. Montgomery, P. M. Chaplya, C. S. Snow, S. J. Glass, R. M. Ferrizz, G. W. Wellman, J. V. Wolf, S. B. Rohde, R. A. Burr, B. S. Altman, T. D. Osborn*

## Abstract

Our team conducted a study to determine the feasibility of creating an embedded neutron generator health microsensor. A truly revolutionary technology for neutron generator design, manufacturing, and surveillance would be to embed health sensors into an operational component. Such technology would enable: (1) validation of current analysis models used for design, (2) real time product quality assessment during manufacturing, and (3) quantitative assessment of aging during long term surveillance. Current advances in microelectromechanical system (MEMS) sensors and wireless interrogation can be utilized to integrate these capabilities into the neutron generator designs of the future. The study team integrated subject matter experts from across Sandia to assess the critical performance parameters that need to be measured as well as the sensor options to develop. This work is setting the groundwork for focused, high risk, and high payoff development of an embedded health monitoring system in neutron generators.

## Accomplishments

Neutron generators are an integral part of nuclear weapon systems, and their function is critically important to those systems. Overall weapon system reliability requires that the reliability of these neutron generators be as high as possible and that the physical phenomena involved in their operation be intimately understood. The high reliability prediction of current neutron generators is based mostly on qualification testing of parts and subassemblies, entire system functional tests to measure neutron output, and discrete aging studies. To help meet the demands of a responsive infrastructure that can successfully develop and manufacture a novel design in a short time span, a fundamental understanding of processes and materials is critical. Utilizing embedded health microsensors to measure critical system parameters is an attractive method to help gain a complete understanding of the myriad of details (processing steps, materials, design objectives, etc.) that produce a robust and reliable neutron generator.

The first major task in this project was to identify performance parameters that are critical to evaluating the health of a neutron generator. A team of subject matter experts helped to identify these parameters and to prioritize them according to their worth for evaluating generator health. These experts identified approximately 40 parameters. This list was corroborated by other sources, including reports in the literature on various subjects, funding for current projects related to neutron generators, and various other resources.

We also asked the team of subject matter experts to identify their highest priorities from the complete list. The majority identified the internal environment (e.g., pressure) to be the most critical attribute for health monitoring. Stress and chemical composition of internal components and overall neutron generator environments complete the identification of the highest priority parameter categories.

The second major task was to identify sensor technologies that have the potential to measure the phenomena from the first objective. The primary focus was on making use of MEMS technologies. We assembled another team of subject matter experts to identify potential sensor technologies and to evaluate the potential readiness of these technologies for the application. They identified a large list of technologies. Two potential sensors with high potential are the ion gauge for measuring pressure and the integrated shock sensor for measuring overall environments. Both of these technologies make use of components already in the neutron generator to make measurements. The sensitivity of the ion gauge approach for accurate pressure measurements is still largely in question, and the technology does not allow measurements during operation of the neutron generator. The integrated shock sensor is based on using the piezoelectric effect of internal components to measure environmental shocks. Many commercial shock sensors use the same phenomena for measurements. There is currently ongoing work to determine the sensitivity of this method and its applicability to the environments that neutron generators are typically exposed to.

The groundwork in this project will allow for more focused, high risk, and high payoff work to be conducted to develop this new capability in the future. The continuous measurements and high measurement density that result from embedded sensor technologies have the potential to significantly improve validation of current analysis models used for design, real time product quality assessment during manufacturing, and quantitative assessment of aging during long term surveillance.

## Refereed

E. Altunok, M.M. Reda Taha, J. Lucero, D. Epp, R.L. Mayes, and T.J. Baca, "Damage Pattern Recognition for Structural Health Monitoring Using Fuzzy Similarity Prescription," submitted to *Journal of Computer Aided Civil Infrastructure Engineering*.

# Phased Antenna Arrays for JTA Systems

*79873*

*K. J. Condreva, M. D. Sena, M. A. Forman, W. E. Patitz, L. A. Forman, R. J. Punnoose*

## Abstract

A typical JTA (joint test assembly) flight test unit utilizes two conformal, omnidirectional antennas mounted around the waist of the outer shell of the test unit. This antenna configuration yields approximately unity antenna gain at most orientation angles between the test unit and the receiving station, except at specific angles where nulls in the antenna gain occur. Since JTA flight test units typically spin, these nulls can cause significant (> 30 dB) fluctuations in the received signal strength. Researchers attempted to address this problem in the past by utilizing both higher power transmitters and novel encoding techniques that typically yield 2–4 dB improvements in link margin. This project is investigating the use of two novel techniques (electronic phasing and frequency diversity) to determine if the nulls in the antenna pattern of typical two antenna JTA systems can be effectively mitigated. This project is also investigating the use of a four antenna array with electronic phasing to determine if further mitigation of antenna nulls is possible. The first phase of this project involved measurement of antenna patterns, from both a single antenna configuration and a typical two antenna JTA configuration, to gain insight into the amount of null mitigation possible. The second phase of the project involves developing prototype telemetry transmission system hardware to demonstrate performance of the techniques under investigation. This demonstration would be accomplished by ground testing of a representative JTA re-entry vehicle in an anechoic chamber. The final phase of the project involves demonstration of compatibility with a commercial satellite system. Since nulls in current JTA antenna systems can produce loss of signal power greater than 30 dB, mitigation or elimination of these nulls would have a huge impact on system performance. For this reason, success of this project could provide a major breakthrough in telemetry system performance.

## Accomplishments

We completed measurements of antenna patterns from both a single antenna and a typical two antenna JTA configuration utilizing Sandia's anechoic test chamber facility. Also, utilizing the results of these antenna pattern measurements, we performed extensive modeling and simulation on various JTA antenna configurations. We used these simulation results to both predict antenna phasing performance and to select the optimum telemetry transmission system hardware design concepts. Work is ongoing to determine if any data reception issues might exist in a JTA implementation of either of the techniques under investigation utilizing typical National Test Range receiving assets. We also began design of prototype telemetry transmission system hardware to demonstrate performance of the techniques under investigation. We completed JTA antenna pattern measurements and completed analyses of two and four antenna arrays.

# Ion Neutron SIMulation—INSIM

*79874*

*G. Vizkelethy, H. P. Hjalmarson*

## Abstract

Electronic components in our strategic nuclear weapons need to survive short intense neutron bursts from hostile countermeasures. These parts must be certified for predetermined radiation levels. In the past, pulsed nuclear reactors, including the Sandia Pulsed Reactor (SPR III), were used for certification. Recently, the availability of pulsed nuclear reactors drastically decreased: the Army Pulsed Reactor Facility (APRF) at Aberdeen Proving Grounds closed, SPR III will be shut down permanently, and the White Sands Missile Range (WSMR) Fast Burst Reactor facility's future is uncertain. Furthermore, new neutron threat regimes have been identified that these reactors cannot simulate.

We aim to continue the development of our ion-neutron simulation (INSIM) technique to use high energy heavy ions to simulate the damage due to short intense neutron pulses. This technique is a fast, inexpensive, and very flexible (duration and intensity of the pulse can be easily varied on wide scale) method to study and validate models of the time-dependent response of transistors to short intense neutron pulses. Furthermore, there are no radiation concerns (activation, personal doses during experiments, etc.) using ion beams. We demonstrated that this technique can be used to prepare and optimize experiment setups for actual neutron experiments. We will compare the results of the ion beam irradiation to actual neutron data and to results of simulation from continuum codes.

We are developing a technique that is not yet proven. We are including a simulation effort that concentrates on the underlying basic physical process and is not geared toward specific devices.

## Accomplishments

We finished the first upgrade of the INSIM system. We modified the deflection system to improve its performance. We determined design criteria for the new ion source to improve the ion flux, and acquired the hardware. We performed several minor modifications to the vacuum chamber to be able to test more than one transistor at a time and to reduce the noise of the system.

We performed Monte Carlo calculations using binary collision codes to determine damage equivalency between different ions and neutrons. We performed preliminary calculations to determine if clustering is significantly different in case of heavy ions and neutrons.

We completed the first round of transistor testing. The goal of these experiments was to determine the effects of the photocurrent (which is significantly higher in the ion beam experiments than in the neutron test) on the measurement electronics, and to find an optimal setup with the highest signal to noise ratio. We found an optimal experimental setup that not only produced data with high signal to noise ratio, but allows us to get reliable data much sooner after the pulse than before. This experimental setup will be used in future neutron tests.

After establishing the optimal experimental setup, we performed late time gain degradation measurements on 2N2222 npn bipolar transistors using several ion-energy combinations. We measured the gain degradation as a function of ion fluence and compared it to the theoretical prediction. As predicted, we found that the differential inverse gain degradation is linearly proportional to the ion fluence for heavy ions (28 MeV Si, 48 MeV Si, and 70 MeV I). On the other hand, we found significant deviation from the linear behavior for light ions (3.9 MeV H and 12 MeV He) at high fluences. Generally, the experimental damage ratios agreed within 15 to 20% with the theoretical predictions.

We performed simulations of transient annealing using the REOS code. These simulations assume a short burst neutron pulse that generates interstitials, vacancies, and divacancies. We obtained the parameters governing the annealing from the literature.

We compared the simulations with transient annealing data for solar cells taken at two different injection rates. We calculated the recombination rate as a function of time for three different injection rates. Data included contributions from the three species: vacancies, interstitials, and divacancies. The more rapid change of the rate, as the injection rate increases, follows from the more rapid annealing of the interstitials. This effect is in qualitative agreement with the effect of injection rate seen in the two sets of data. Furthermore, the injected carrier density is similar for the

theory and the experiments. Further work is needed for this assessment because the carrier density depends on how much annealing has occurred.

The main contribution to the recombination rate at short times is due to interstitial recombination. The evolution to shorter times with increasing carrier injection is due to the increased athermal diffusion stimulated by the carrier recombination at the interstitials. The time scale of vacancy evolution is nearly independent of injection rate. At the highest injection level, substantial interstitial annealing occurs before vacancy annealing. Thus the vacancy contribution shows up as a plateau in that simulation.

Comparing the theory with the data reveals that the data have a more gradual evolution than that seen in the calculations. This may be due to contributions from competing processes not included in this minimal simulation. It may also be due to complications from effects that occur in the diodes used in the experiments that are absent in the bulk simulations. This issue will be examined in future simulations.

# A Miniaturized mW Thermoelectric Generator for NW Objectives: Continuous, Autonomous, Reliable Power for Decades

*79875*

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

## Abstract

We developed a new, miniature thermoelectric device that will produce 100 µW of power from a 150 mW $^{238}$Pu source. A simulated electrical power pellet was fabricated to take the place of the nuclear material for testing. Early attempts to build a planar geometry device showed that very little heat from the source was being delivered down the legs of the converter, resulting in very little power generation. A new architecture, based upon a radial geometry, was built and tested, and showed an initial power of 10 µW. With improvements to the contact resistance that we developed, this power source should produce 100 µW. Several predictive models have been developed that show where the remaining problems to power generation lie. Future efforts will focus on improvements to eliminate these problems.

## Accomplishments

We developed and tested three prototypes, and developed numerical models of each prototype. The first prototype used a silicon membrane based approach with thin film materials bridging between the center hot zone and edge cold zone. However, this prototype did not generate any power, due to high radiation of heat from the top and bottom surfaces. Once the heat flow problem was understood, we developed an annular prototype design that demonstrated the first power of 10 µW against a load, but this prototype suffered from high internal resistance. This contact related resistance problem has since been solved, and the new prototype is expected to generate 110 µW of power. A final problem relates to the loss of heat through the top and bottom of the device, and new materials with low thermal conductivity are being built that should significantly increase the heat flux through the thermoelectric converters. With these three improvements, the power from the device should be approximately 350 µW. Further improvements in minimizing the power lost from Peltier effects and increasing the output voltage should reasonably be expected to push the power up past the 1 mW level.

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

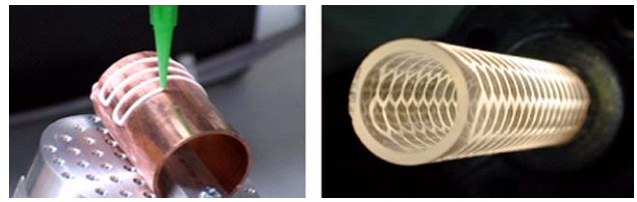
*79876*

*J. Cesarano III, A. S. Tappan*

## Abstract

Recent advances in fabrication technology coupled with availability of energetic nanomaterials present a tremendous opportunity for interdisciplinary research spanning the fields of precision free form manufacturing, and energetic materials. We will advance the state-of-the-art for three-dimensional (3-D) printing of materials to allow conformal printing of multiple materials in graded compositions. We will apply these fabrication improvements, based on Sandia's robocasting technology, to the manufacture of precision-deposited and free-form energetic bodies. The starting materials include nanoscale powders of metals, oxides, ceramics, and explosives. We will control macrostructure with microprecise, 3-D robocasting, and the material micro- and nanostructure will be defined by the combustion synthesis. In the first application, laboratory controlled combustion synthesis will form an advanced material. This could be an advanced alloy such as Nitinol from the combustion synthesis of Ni/Ti, an advanced metal-ceramic such as Al/$TiB_2$ from the combustion synthesis of Ti/B/Al, a ceramic, or a functionally graded material composed of different materials. In the second application, the advanced material will be combustion synthesized during function of a device. This will be useful when a dynamic material and/or property transition is necessary for functionality. The material/property transition may be initiated with an applied signal, a temperature change, or a mechanical shock.

Initially, proof-of-concept demonstrations of both applications will be conducted, with later efforts involving refinement coupled with combustion and material modeling. This project will create new functional materials not available by other fabrication or synthetic techniques and will provide a suite of materials and applications rather than one specific to a programmatic need. We anticipate additional specific applications to present themselves as the project progresses. Rapid prototyping of different materials/configurations will be possible with this technology, allowing facile application to varying needs as they arise. This project will impact material science, surety applications, and weapon systems where structural energetic components can increase energy density.



(left) *A fourth axis of motion control was incorporated to allow deposition on a cylindrical surface while keeping the tip normal to the print surface.* (right) *A pattern resembling the image of a nanotube was successfully drawn on a cylinder.*

## Accomplishments

The overall goal of this project is to couple free-form fabrication and precision-deposition with energetic materials for applications requiring irreversible changes in properties. We want designers to have a deposition tool that allows them to integrate energetic materials into components for improved surety and safety. There are two types of applications/material systems of interest: (1) harvesting the products of energetic materials, i.e., heat, light, pressure, etc.; and (2) materials that (when initiated) function as reactants for self-propagating high temperature synthesis to produce different materials.

We primarily focused on establishing the feasibility of coupling precision deposition methods (i.e., robocasting) with energetic materials, demonstrating the functionality of cast materials, and advancing precision deposition technology, in general. More specifically, we accomplished all our objectives, including down selection of materials, development of castable slurries of these materials, testing, and advancement of robocasting technology.

We developed a casting slurry of Al/$Fe_2O_3$ and Al/Ni that exhibited the required rheology suitable for robocasting nonslumping structures. We developed the capability to formulate slurries of energetic materials with all requisite safety controls in place. We demon-

strated the robocasting of two energetic materials systems, $Al/Fe_2O_3$ and Al/Ni, and subsequently initiated the cast materials. We demonstrated the capability to cast on nonplanar surfaces by performing laser profiling and motion-control programming. We enhanced the capabilities of robocasting with the addition of a fourth axis, allowing pen-normal casting on cylindrical substrates that will enhance resolution and conformal coating precision. We also demonstrated the capability to cast mock materials with the appropriate viscosity to learn how to deposit ultrahigh energy materials. We identified numerous applications during discussions with other groups at Sandia.

## Refereed

J. Cesarano, "Direct-Write Fabrication with Fine Particle Suspensions," presented at the Materials Science & Technology Meeting, Pittsburgh, PA, September 2005.

# Surety Enabled by Reduced Cost

*86364*

*R. Wild, E. J. Welle, W. H. Greenwood*

## Abstract

Perhaps the biggest roadblock to substantial increases in weapon system surety is the cost of providing increased functionality. Surety mechanisms play a major cost role in components such as modern unique signal (UQS) stronglinks that can typically require $30,000 to produce prior to final testing and inspection. Various attempts over the years to reduce surety mechanism cost through incremental improvements and design for manufacturability studies have had only a small (less than 10 percent) impact on cost.

The purpose of this project is to investigate a lower cost electrical stronglink (LESL) that would provide the functionality of an intent stronglink (i.e., intent unique signal (IUQS) discrimination, monitoring, lockup, enabling) in a volume of about 1.13" x 1.9" x 1.6". It will provide three energy coupler channels (i.e., power, data, and ground) with a unique signal start to enable operation time of about three seconds.

The LESL will have a one time premission “state-of-health” function that will provide a confirmation that the stronglink mechanism is functional. After the state-of-health test, a monitor will detect that the discriminator is ready to receive the UQS.

## Accomplishments

The LESL’s computer aided design layout is near completion.

We drafted a compatibility document for the lower cost magnetic stronglink, and we began its computer aided design layout. The concept review went well; however, we must still complete the monitor design concept.

# Advanced Manufacturing

## Atomic Layer Deposition of Highly Conformal Tribological Coatings

*52523*

*T. M. Mayer, M. T. Dugger, T. W. Scharf, R. S. Goeke, N. R. Moody, S. V. Prasad, R. K. Grubbs, J. M. Jungk*

### Abstract

Friction and wear are major concerns in the performance and reliability of microelectromechanical systems (MEMS) devices. While a variety of lubricant and wear resistant coatings are available for application to MEMS devices, the severe geometric constraints of many micromechanical systems (ex. high aspect ratios, shadowed surfaces) make most deposition methods for friction and wear resistant coatings impossible. In this project we have produced and evaluated highly conformal, tribological coatings deposited by atomic layer deposition (ALD), for use on surface micromachined (SMM) and LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) structures.

ALD is a chemical vapor deposition process that uses sequential exposure of reagents and self-limiting surface chemistry, saturating at a maximum of one monolayer per exposure cycle. The self-limiting chemistry results in conformal coating of high aspect ratio structures, with monolayer precision. ALD of a wide variety of materials is possible, but there have been no studies of structural, mechanical, and tribological properties of these films. We developed processes for depositing thin (< 100) nm) conformal coatings of selected hard and lubricious films ($Al_2O_3$, ZnO, $WS_2$, W, and W/$Al_2O_3$ nanolaminates), and measured their chemical, physical, mechanical, and tribological properties. A significant challenge was to develop nonexisting instrumentation and quantitative test procedures for friction, wear, film/substrate adhesion, elastic properties, stress, etc., of extremely thin films and nanolaminates. New scanning probe and nanoindentation techniques were employed along with detailed mechanics-based models to evaluate these properties at small loads, characteristic of microsystem operation. We emphasized deposition processes and fundamental properties of ALD materials; however, we also evaluated applications and film performance for model SMM and LIGA devices.

### Accomplishments

This project addresses the issue of lubrication and wear resistant coatings on MEMS. Friction and wear are major concerns in the performance and reliability of MEMS devices. While a variety of lubricant and wear resistant coatings are available for application to MEMS devices, the severe geometric constraints of many micromechanical systems (ex. high aspect ratios, shadowed surfaces) make most deposition methods for friction and wear-resistance coatings impossible. In this project we have produced and evaluated highly conformal, tribological coatings deposited by ALD for use on SMM and LIGA structures.

ALD is a chemical vapor deposition process that uses sequential exposure of reagents and self-limiting surface chemistry, saturating at a maximum of one monolayer per exposure cycle. The self-limiting chemistry results in conformal coating of high aspect ratio structures, with monolayer precision. ALD of a wide variety of materials is possible, but there have been no studies of structural, mechanical, and tribological properties of these films. We developed processes for depositing thin (< 100 nm) conformal coatings of selected hard and lubricious films ($Al_2O_3$, ZnO, $WS_2$, W, and W/$Al_2O_3$ nanolaminates), demonstrated the ability to coat very high aspect ratio structures with these materials, and measured their chemical, physical, mechanical, and tribological properties. We devoted major attention to deposition and characterization of

three materials. $WS_2$ is a superior solid phase lubricant, W is a hard metallic, wear resistant material, and W/$Al_2O_3$ laminate films are potential "superhard" coatings with properties determined by the nanometer length scales of the laminate layers.

We developed a deposition process for $WS_2$ using $WF_6$ and $H_2S$ precursors in a laminar flow ALD reactor. We found growth to be limited by adsorption and decomposition of $WF_6$ on most surfaces, and a novel Zn catalyzed deposition method was identified. We propose that $WF_6$ is partially reduced by Zn or ZnS on the surface, and periodic introduction of $(C_2H_5)_2Zn$ greatly enhances the growth of $WS_2$. Tribologial measurements of $WS_2$ films have shown them to have very low friction coefficient, approx. 0.02. Coatings only tens of nanometers thick can give low friction and increase wear life since sliding is interfacial, i.e., between the transfer film and the wear track on the coating. The hexagonal crystal texture of ALD $WS_2$ is temperature dependent, but shear-induced reorientation at our high contact stresses assures low friction.

W and W/$Al_2O_3$ films grown at the University of Colorado are also shown to be limited by nucleation of W from $WF_6$, and extensive studies of W nucleation detailed the growth process. Very uniform, smooth films of W and laminates result from proper control of nucleation kinetics.

Extensive mechanical property measurements of all ALD films measured detail of the modulus, hardness, interfacial strength, and crack and delamination mechanisms for these films. Laminate film properties were shown to be dependent on the length scale of the individual film thicknesses in the laminate, reflective of the effects of interfaces on the deformation and failure properties of these materials.

A significant challenge was to develop non-existing instrumentation and quantitative test procedures for friction, wear, film/substrate adhesion, elastic properties, stress, etc., of extremely thin films and nanolaminates. We employed new scanning probe and nanoindentation techniques with detailed mechanics-based models to evaluate these properties at small loads, characteristic of microsystem operation. We emphasized deposition processes and fundamental properties of ALD materials; however, we also evaluated applications and film performance for model SMM and LIGA devices.

## Refereed

T.W. Scharf, S.V. Prasad, T.M. Mayer, R.S. Goeke, and M.T. Dugger, "Atomic Layer Deposition of Tungsten Disulfide Solid Lubricant Thin Films," *Journal of Materials Research*, vol. 19, p. 3443, 2004.

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N. 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 Films," presented at the European Conference on Fracture, Alexanderoupolis, Greece, July 2006.

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T. Scharf, S. Prasad, M. Dugger, P. Kotula, R. Goeke, T. Mayer, and N. Moody, “Growth, Structure, and Tribology of Atomic Layer Deposited Hard and Solid Lubricant Coatings,” presented at the International Conference on Metallurgical Coatings and Thin Films, San Diego, CA, May 2005.

T. Scharf, S. Prasad, M. Dugger, T. Mayer, R. Grubbs, and R. Goeke. “Atomic Layer Deposition (ALD) of Tribological Coatings,” presented at the Conference on Advanced Ceramics and Composites, Cocoa Beach, FL, January 2005.

R. Wind, F. Fabreguette, and S.M. George, “The Complex Nucleation Behavior of W ALD on $Al_2O_3$ Revealed by Quartz Crystal Microbalance Measurements,” presented at the Conference on Atomic Layer Deposition 2005, San Diego, CA, August 2005.

# Assembly of Microsystems Using Exothermic Multilayer Thin Films

*52524*

*D. P. Adams, F. M. Hosking, T. E. Buchheit, C. P. Tigges, M. A. Rodriguez*

## Abstract

This project researches properties of energetic thin films and new assembly techniques involving these thin film coatings. Energetic films consist of two or more reactive materials typically deposited as a heterostructure. As previously demonstrated, properly designed layered materials can be stimulated in a local region to cause a self-propagating exothermic reaction (alloying) over great distances or simultaneous combustion of reactants. The heat evolved from this reaction can be used to drive reactions and to join dissimilar components.

We are researching a number of properties in order to improve the manufacturability of this process, including research of fundamental material properties such as combustion temperature and its dependence on substrate thermal diffusivity, film design, and film thickness. Furthermore, we are researching several new energetic material pairs (not reported within the literature) to assess their usefulness for joining. We are investigating the effects of a high speed reaction on product phase, microstructure, and mechanical properties.

We are investigating methods for precision machining of exothermic films after removal from the deposition system. Most components to be joined are planar, but many require odd shapes. For example, sealing along the periphery of a package lid may require a preform. Our current research investigates femtosecond pulse width lasers for precision machining. Ultra-short pulse lasers have a minimal heat affected zone around the irradiated area, and this potentially allows for removal of material without ignition.

## Accomplishments

We researched the properties of more than ten energetic thin film systems, including Co/Al, Ni/Ti, and Al/Pt. Studies include analysis of steady-state propagation speed dependence on multilayer design, microstructure and product phase formation, influence of substrate thermal properties on propagation, mechanical properties of assembly and reacted film, and the ability to laser machine energetic films into various preform shapes.

We demonstrated successful joining methods for assembly at the micro- and mesoscales. This was accomplished using optimized Co/Al energetic thin films that react when constrained between two parent materials. Low-temperature joining by this material pair had not been previously reported in the literature. Mechanical properties of joined assemblies were analyzed by tensile testing.

Co/Al is preferred over all other tested systems. It is reactive for different multilayer designs having propagation speeds ~10 m/s. Additionally, it forms a B2 structure for all films tested. This is beneficial since this phase exhibits moderate ductility, thought to be critical for accommodating the extrinsic stress upon cooldown from the combustion temperature. Other candidate energetic pairs have limited use for joining because of low heats of formation and other factors. For example, Ni/Ti can form a B2 structure but is compromised by the formation of a monoclinic B19' phase.

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# Intelligent Interaction Control as Applied to Metrology and Assembly of Microscale Components

*52526*

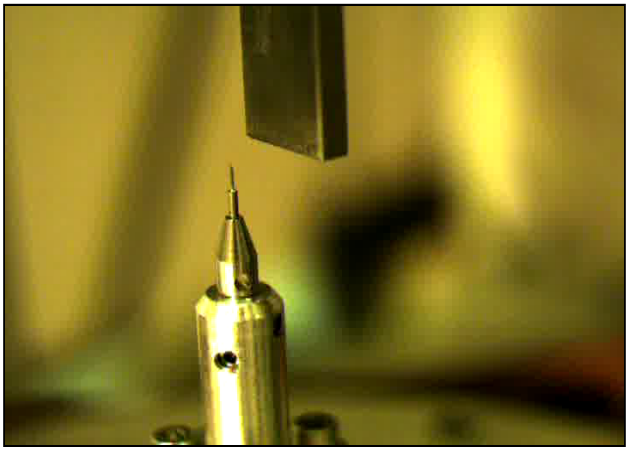
*D. M. Kozlowski, J. F. Jones, N. A. Weir, D. G. Wilson, W. J. Prentice, A. L. Ames*

## Abstract

Dimensionally inspecting microscale parts is extremely difficult. Current technology uses microscopes to optically magnify the part and image processing to extract the dimensional relation between features. Optical measurement techniques have limitations in microscale applications for two reasons. First, they are effectively a two-dimensional process, meaning that only the surface of the part is measured and very limited information concerning the sidewalls of high aspect ratio parts is obtained. Second, optical measurements are physically limited to about one-micron resolution, and practically limited to about two-micron resolution. It would be highly desirable to have a cost effective way to dimensionally inspect production quantities of microscale parts with submicron resolution.

Hard gauging is a common macroscale process because it is a cost effective means to validate functional dimensions at production rates. A hard gauge typically consists of a precision fixture that holds the part and a variety of go/no-go "feelers" used to verify maximum and minimum material conditions. Using hard gauges, it is straightforward for a human technician to obtain 10 to 20 micron (e.g., less than one thousandth of an inch) validation of part dimensions. The limiting factor is the human's ability to detect the variation in force required to insert the "feelers," hence the origin of the term "feeler gauge."

During FY 2005, we integrated a high precision Gough-Stewart nanomanipulator, combined with advanced position and force control algorithms, into a research testbed toward a hard gauging conceptual solution. Utilizing precision gauges, the current research shows the capability to go beyond human sensory perception in controlling forces at the microscale (with the limiting factor being the multi-axis force sensor capability). We performed initial validation experiments comparing the actual part parameter dimension with the dimension measured using the proposed concept.



*Movie of a system tracing gauge under force control.*

## Accomplishments

During FY 2005, we completed a research and development testbed using a high-precision manipulator, advanced controls, and custom software in order to ascertain the viability of dimensional inspection at the microscale utilizing a hard-gauging concept, similar to human macroscale approaches (also known as "feeler" gauging). We integrated an ultra-high precision Gough-Stewart robotic nanomanipulator (a parallel robotic platform, extremely stiff and rigid with excellent strength to weight ratio, and much more accurate when compared to serial link robotic manipulators) with advanced control algorithms into a micro-gauging testbed. We conducted two different methods of testing. The first method utilized the accuracy of the platform together with advanced force control operating in a hybrid control scheme (position and force control operating on prescribed axes). We used a precision Ruby ball as an end-effector to measure the sizes of various precision hard gauges and hole sizes of some LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) devices by tracing along the part in question. Although the error in the kinematic model of the manipulator increased error in the measurement, this method made for an excellent starting metric for the system. Utilizing the gauges, a plane was fit for a por-

tion of each side of the gauge. Residual errors for each plane indicated errors less than 0.2 micron. The second method utilized the same end-effector with the same fundamental control to measure the gauging process by insertion of a precision Ruby ball of known diameter into a hole of a known diameter. This is similar to a “peg in the hole” problem, but with tolerances on the order of microns. We obtained successful “feeler” gauging after determining the normal vector to the desired insertion (required, or else jamming occurred).

# Rapid Prototyping High Density Circuitry (RpHdc)

*52527*

*D. W. Davis, J. M. Sena, S. C. Anderson, P. G. Clem, J. A. Palmer, B. D. Chavez, J. M. Kelton, P. Yang*

## Abstract

A new manufacturing process has been developed to integrate complex modeling, rapid prototype stereolithography processes, and direct-write technology for the fabrication of three-dimensional (3-D), high-density circuits. This paperless manufacturing technology provides greater flexibility for rapid development and deployment of new designs, especially for systems with weight and space restrictions.

The research for this project will be conducted along two intertwined paths: materials and process. The materials path will be guided by both the inherent user requirements (physical and chemical compatibilities with different materials in the fabrication process, stability of the underlying substrate, conductivity and longevity of the drawn circuit material, etc.), as well as the inherent limitations of stereolithography-compatible photopolymers (low glass transition temperatures, mechanical properties, etc.). The process studies will revolve around techniques to start, stop, fixture, and reregister stereolithography builds, as well as the mechanics of depositing the circuit materials onto the layer-manufactured substrates. Techniques to embed sensors, create multi-layer embedded circuits, and provide robust interconnects will also be studied.

This new technology will provide for the direct writing of high density conductive paths directly onto, and embedded in, the layers of the complex model. This technology will eliminate many of the labor intensive tasks currently required during the assembly process. This technology will also reduce the hardware required to interconnect assemblies and will combine known technologies to develop a single electrical/mechanical "design to manufacturing" process in support of rapid development and deployment of fully functional prototypes as the final product.

## Accomplishments

We continued our partnership with the University of Texas at El Paso (UTEP) to fabricate the stereolithography apparatus (SLA) parts made with the DSM Somos ProtoTool 20 L resin. We submitted two patent applications as a result of this partnership: "Methods and Systems for Rapid Prototyping of High Density Circuits" (SD-7758/application #10/907,979) and "Methods and Systems for Integration of Thermally Curable Media During Stereolithography" (SD-7813/application #11/905,120).

- FY 2005 Milestone 1 – Continue Development of Complex Part Fabrication

The team selected a portion of their Protocol Converter to demonstrate using the RpHdc process. A 28 to 5 volt direct current (DC) to DC converter, connector, electromagnetic interference (EMI) filter, and light emitting diode (LED) were embedded into the stereolithography structure. Part registration parameters were established and the base structure built. The stereolithography part was then removed and the electronic devices placed into the structure. The part was remounted in the stereolithography machine, and the layers were built up to cover all but the pins of the DC to DC converter and the EMI filter. The part was then sent to the Direct Write machine and the first layer of Direct Write traces was written. The subsequent build layer was added, including a "well" area around each of the contact points on the ramp. These "wells" provided the means for the conductive ink to pool and connect the bottom layer of the part to the top layer of the part.

We subjected the part to 50 cycles of thermal cycling between -40 and +85 °C. The LED failed during the thermal cycling and was replaced. The RpHdc part survived the thermal and 25 g shock cycling and is a functioning unit.

- FY 2005 Milestone 2 – Produce 3-D complex models with 1 mil lines and spaces

Our goal of 1 mil lines and spaces was not achieved on 3-D stereolithography parts. The smallest traces that we were able to write on the 3-D SLA material were 3 mil lines and 1 mil spaces. These lines were written using the Paralec SDA-301 Silver High Conductivity Ink. It is a challenge to write 1 mil lines on the rough surface generated from the SLA build process in a 3-D part. The areas on the non-planar part that are planar and that were manufactured horizontal to the SLA machine bed do provide a smooth surface on which sub 2 mil lines can be written. The team was able to write 1.1 mil lines with 1 mil spaces on planar

ceramic material with the same SDA-301 ink.

- FY 2005 Milestone 3 – Determine if the electrical and mechanical designs can be incorporated into a single software design package

To date, the team has not been able to directly use files generated in the 3-D mechanical model for direct use in generating the electrical portion of the design. The difficulty lies with the Direct Write machine using a 2-D drawing exchange format (DXF) file for its writing pattern. The team is still pursuing this milestone and has some new ideas to investigate. The team believes there are still a couple of possibilities using the SolidWorks 3-D Modeling software.

## Other Communications

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# Elucidating the Mysteries of Wetting

*52528*

*G. S. Grest, A. M. Grillet, P. A. Sackinger, C. F. Brooks*

## Abstract

Nearly every Sandia owned manufacturing responsibility and many technologies central to Sandia's business involve physical processes controlled by interfacial wetting. Interfacial forces (such as conjoining/disjoining pressure, electrostatics, and capillary condensation) are omnipresent and can surpass and even dominate bulk inertial or viscous effects even on a continuum level. Moreover, the statics and dynamics of three-phase contact lines can exhibit a wide range of extraordinarily complicated behavior, such as contact angle hysteresis due to surface roughness, surface reaction, or compositional heterogeneities. These statics and dynamics all manifest themselves in unpredictable ways when attempting to control liquid/gas motion on length scales. These thermodynamically and kinetically driven interactions are essential to the development of new material systems such as nanocomposites as well as the fabrication of complex microassemblies.

The focus of our research has been to advance existing theoretical/computational tools and approaches and to develop pertinent experimental diagnostics to analyze systems and processes dominated by capillary hydrodynamics effects. This involved a three-pronged approach, combining atomistic and continuum level modeling with experimental studies. During the three years of the project, new experimental tools and diagnostics were implemented to measure concentration segregation during wetting. The atomistic simulations concentrated on determining the wetting properties of multi-component mixtures of polymer fluids for different chain lengths and equilibrium contact angles, and comparing the spreading dynamics to existing continuum models. The continuum approach has been used to investigate several models for droplet spreading.

## Accomplishments

Experimental capabilities were developed to examine the wetting behavior of multi-component polymer blends. Starting with polybutadiene adducted with maleic anhydride, the polymer chains were labeled with various fluorescent tags using synthetic chemical methods. By varying the molecular weight of the polybutadiene and choosing fluorescent tags that excite/emit at different wavelengths, a matrix of mixtures was established and used to develop fluorescence microscopy methods. To characterize polymer blend properties, a modified Wilhelmy probe technique was implemented for determining the liquid surface tensions of small amounts of polymer blends. The surface tension of a blend was found not to obey a simple linear mixing rule. Rather, a dramatic drop in overall surface tension was observed even at low mole fractions of the second component due to surface segregation of the lower surface tension component. Using a digital video system and an interface-fitting program, a continuous feed-through goniometer was built to study the contact angles and drop shapes during dynamic wetting.

These experimental tools were applied to study the wetting behavior of multi-component blends of low molecular weight polymers. A series of polypropylene glycol polymers of varying molecular weights but very similar surface tensions was studied. For both fully wetting and partially wetting cases, the wetting of the blends was determined to obey a simple scaling rule based on the blend viscosity. More complex blends of polypropylene glycols, with either polyethylene glycols or random copolymers of polyethylene and polypropylene glycols, were also investigated. The wetting behavior of these materials was also more complicated since the surface tensions and equilibrium contact angles of the individual components were different, and no simple mixing rules for the behavior of the blends were apparent with the limited data available. All of the experimental spreading data were successfully fitted using several continuum models for dynamic contact angles, including Blake's molecular kinetic model and the Seaver-Berg hydrodynamic model.

Previous molecular dynamics simulations of polymer droplet spreading were extended to include binary systems where the components vary in either the polymer chain length or the surface interaction strength. An investigation of the qualitative spreading behavior of binary droplets and a detailed quantitative analysis were carried out. Large droplets on the order of 1 mil-

lion monomers were simulated to study the dependence of the kinetics on droplet size. We found that the precursor foot spread diffusively with an effective diffusion constant that scaled with the square root of the initial droplet radii. The volume dependence of the main droplet was consistent with the predictions of the kinetic model of droplet spreading. By using analytical models of droplet spreading based on surface adsorption/desorption and viscous flow mechanisms, we were able to demonstrate the presence of hydrodynamic dissipation for the first time in molecular simulations of spreading droplets. We gained insight into the source material for the precursor foot and the extent of component intermixing in the bulk region of the droplet by analyzing the instantaneous velocity distribution throughout the droplets during the spreading simulations.

We also developed and implemented algorithms for successfully simulating moving contact lines for continuum formulations of the Navier-Stokes equations in Sandia's production finite element program GOMA. Adding to an existing capability for simulating transient non-linear momentum transport in deforming fluid domains with capillary normal stresses, we discovered a robust means for applying the distinguishing constraint conditions that apply to the three phase line. Three constitutive models were implemented to describe the relation between contact line velocity and the apparent contact angle, including a kinetic model, a linearized variant, and a general power law model that embodies Tanner's law. Continuum simulations of drop spreading, driven solely by wetting forces at the three phase line, compared well with experimental results. Our computations provided insight into the partition of spreading power into bulk viscous dissipation, and into slipping dissipation for subgrid models of contact line dynamics.

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# Rapid Prototyping to the Nanometer Scale

*67004*

*D. P. Adams, G. R. Burns, T. M. Mayer, M. J. Vasile, M. B. Sinclair*

## Abstract

Commercial rapid prototyping (RP) techniques are currently limited to large dimensions (greater than millimeters) and are restricted in most cases to polymers. In this project, we research and develop new RP techniques that cover 30 nm to 1 mm length scales in a diverse set of materials.

Our research takes advantage of focused ion beam (FIB) methods for defining features in virtually any solid material. With beam widths as small as 20 nm, FIB sputtering and FIB-induced chemical vapor deposition are useful for defining 0.03–250 micron wide features. In order to control the depth and three-dimensional (3-D) shape of an ion-milled feature, we couple FIB methods with two in situ metrology techniques.

We are researching in vacuo interferometry for shape profiling, end-point detection and closed-loop control. Custom optics will provide 1 micron in-plane and 5 nm depth resolution. The design of vacuum-compatible, long working, distance optics is a significant part of the innovation. Interferometry should be capable of probing complex ion-milled shapes. This will be demonstrated by milling a variety of curved shapes including hemispheres and paraboloids into monolithic targets.

## Accomplishments

We designed, fabricated, and tested a vacuum-compatible Michelson interferometer microscope for real-time optical profiling during FIB bombardment. We intended the instrument to have 1 micron in-plane resolution and 5 nm out-of-plane accuracy. We targeted slope information to 25 degrees from normal incidence to allow precise monitoring of curved feature shapes. The interferometer must be noninvasive and should in no way degrade the accuracy of feature definition. The interferometer includes two custom designed, vacuum-compatible imaging objectives oriented at 90 degrees with respect to each other. The system allows operation at any wavelength between 510 and 550 nm (green to yellow).

In FY 2005, we constructed and tested several objectives as part of the interferometer to evaluate overall system performance. Initial benchtop tests with a lithographic master, and follow-on tests with FIB-milled Si specimens, demonstrate ~1.0 micron in-plane resolution. We also examined the out-of-plane resolution using features milled to various depths from 5 to 100 Å. These tests indicate a 5 nm out-of-plane resolution, or better. We determined the optical field of view to be 175 microns.

Our current design translates a sample along the ion beam axis to map the profile of ion-milled features. This is accomplished using an ultra-high-vacuum compatible, nonmagnetic (< 1 nT), hysteresis-compensated, piezoelectric compact z-positioner. We incorporated this stage onto a custom-built, removable sample cassette that has two independent tilt axes for precise alignment of the z travel axis to the ion beam vector. We tested it through the full 250 micron range, and it maintains 5 Å resolution. We determined that sample translation along the ion beam axis will not degrade the quality of a milled feature (i.e., no feature broadening). Experiments used to prove this involved measurement of the FIB depth of focus. Specifically, we ion milled single pixel wide lines using a 700 nm wide beam while maintaining a fixed (lens 2) focal voltage. The specimen was translated in z (parallel to the ion beam vector) to 10 microns after completing each milled line. Afterwards, atomic force microscopy measured feature width. There is no detectable change in ion-milled feature width over a large range of working distances (140 microns). We only detected a deviation in feature size after translating the specimen 220 microns about the optimal focus. This large FIB depth of focus is greater than the typical depth of ion-milled features.

## Refereed

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# Robust Manufacturing of Gel-Based Components for Nuclear Weapons

*67005*

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

## Abstract

The development of non-aqueous polymer gels is of critical importance to the management of the nuclear weapons stockpile because they are the primary risk associated with gel-Mylar capacitors, a key nuclear safety component in ongoing Life Extension Programs. A gel that is suited to this application consists 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. The first year of this project has sought to establish a relationship between the gel fabrication procedure and the resulting mechanical properties, gel microstructure, and phase behavior. To develop this link, gels were prepared using one of two solvents in various proportions. In addition to confirming typical mechanical behavior for crosslinked networks, an impact of solvent content during gel processing was found. Rheological analysis of these gels, in combination with solvent extraction, has shown that the screening of entanglements (physical crosslinks) is changed dramatically by varying the solvent content.

The second year of this project further expanded the exploration of gel microstructure and the impact of solvent. A combination of neutron scattering and rheological characterization has shown that the content and quality of solvent used in the gel fabrication, as well as the content and quality of a swelling solvent, can be used to control the ultimate gel properties. In this way, the final microstructure and properties may be set through the preparation procedure and solvent, which are both controllable. This work will form the scientific foundation necessary to exploit gels for Sandia and Department of Energy applications.

## Accomplishments

All of the milestones for FY 2005 were completed. Rheological characterization techniques have been extended and used to confirm phase separation in dibutylphthalate (DBP)-based gels and homogeneity in bis(ethyl hexyl) sebacate (BEHS)-based gels. Neutron scattering work has further confirmed the differences imparted by these two solvents on the gel microstructure. The temperature range through which phase separation occurs in DBP-based gels has been explored using neutron scattering, and is being used to evaluate the impact of a change in solvent quality on the gel properties. Gels with various effective crosslink densities have been fabricated by varying the solvent content. Rheological analysis and solvent extractions of these gels have provided evidence that the number of effective crosslinks is a combination of chemical crosslinks and physical crosslinks, termed *entanglements*. Using the solvent content to control the screening of entanglements was shown to be an effective method of altering the gel mechanical properties and microstructure. Swelling of these gels with controlled microstructure suggested the possibility of manipulating a series of gel properties through the compatibility of the swelling solvent with the gel network. This will form the basis of a processing strategy to control gel properties and expand the application of gels to other technologies.

# Meso-/Micro-Optical System Interface Coupling Solutions

*67006*

*S. A. Kemme, M. R. Daily, D. K. Serkland, M. Armendariz, T. Carter, S. Samora, R. Boye, R. T. Mitchell*

## Abstract

Optoelectronic microsystems are more and more prevalent as researchers seek to increase transmission bandwidths, implement electrical isolation, enhance security, or take advantage of sensitive optical sensing methods. Board level photonic integration techniques continue to improve, but photonic microsystems and fiber interfaces remain problematic, especially upon size reduction.

Optical fiber is unmatched as a transmission medium for distances ranging from tens of centimeters to kilometers. The difficulty with using optical fiber is the small size of the core (approximately 9 microns for the core of single mode telecommunications fiber) and the tight requirement on spot size and input numerical aperture. Coupling to devices such as vertical cavity emitting lasers (VCSELs) and photodetectors presents further difficulties since these elements work in a plane orthogonal to the electronics board and typically require additional optics. This leads to the need for a packaging solution that can incorporate dissimilar materials while maintaining the tight alignment tolerances required by the optics.

Over the course of this project, we examined the capabilities of components such as VCSELs and photodetectors for high speed operation and investigated the alignment tolerances required by optical systems. We developed a solder reflow process to help fulfill these packaging requirements, and the results of that work are presented here.

## Accomplishments

We investigated the electrical, optical, and mechanical requirements for a passively aligned optoelectronic system and pursued the development of an appropriate packaging technology to facilitate its realization. We characterized high speed components, including VCSELs and photodetectors, and developed models to aid in the design of a fully functional optical link demonstration. The measured VCSEL modulation bandwidth reached 12 GHz. We also developed electrical transmission line models using the diverse materials required for this type of link. We determined that transmission lines capable of 10 Gbps could be developed with a single mask set using several common electrical board materials and fused silica, a common micro-optical material.

We determined the required optical alignments for use with optical fibers and high speed detectors. We modeled a sample system, coupling the output of a single mode fiber to a high speed photodetector. A required lateral alignment of 5 microns and angular alignment of 2 degrees was necessary for 95% coupling efficiency. These mechanical requirements, along with the limitations imposed by the use of dissimilar materials, guided the development of an appropriate bonding process. We pursued indium tin solder, with emphasis on developing a deposition process (that provided the desired eutectic alloy as well as good volume control) and a reflow process capable of providing self-alignment. We pursued four solder deposition methods with successful volume control (uniformity < 5%) achieved using jet vapor and evaporation based approaches. We demonstrated self-alignment with submicron accuracy with a flux-based process that successfully bonded fused silica and a silicon test die.

# Injection Molding of Net-Shape Active Ceramic Components

*67007*

*P. Yang, A. M. Grillet, C. B. DiAntonio, L. A. Mondy, R. R. Rao, G. R. Burns, L. L. Halbleib, R. H. Moore*

## Abstract

We propose studying and utilizing an injection molding technique for the fabrication of active ceramics components used in the power supply unit of neutron generators. The new approach will significantly reduce pressing defects and machining-induced surface defects. Ceramics with surface defects are more susceptible to handling damage in subsequent processes. For example, more than 20% of voltage bars are lost due to cracking and chipping induced during the manufacturing processes. More importantly, by eliminating the machining processes, the net-shape forming approach can save 18% of the touch labor cost, minimize the hazardous waste stream management, trim several processing steps, and make it a true lean manufacturing process.

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 compound, the pressure of material in the cavity, and tooling parameters. To date, the injection molding of ceramics has been essentially experimental in nature. We will develop a finite element analysis capability for the flow modeling to determine the processing and tooling parameters. Furthermore, the control of sintering shrinkage and lead loss during high temperature processing is also critical for material performance. Because injection molding can offer the promise of high volume, low cost net-shape production of micromechanical devices, establishing a knowledge base for ceramic components fabrication will pave the road for future application development.

## Accomplishments

We modeled the kinetics of organic removal by modifying a master sintering curve theory. The “activation energies” of three major events determined by the master curve theory were 82, 68, and 120 kJ/mole at the initial, intermediate, and final stages, respectively. Following the kinetic study on organic removal, we are trying to establish optimized thermal treatment strategies from the master decomposition curve for this system. Additional oxidation processes, due to the formation of carboxylates from polyethylene and polypropylene on the ceramic surface during the dewaxing, were identified using Fourier transform infrared (FTIR) spectroscopy. These results helped us obtain a better understanding of thermal degradation and oxidative degradation processes during the organic removal process.

We used a finite element model to describe the injection molding of the ceramic feedstock. Preliminary calculations, both two-dimensional (2-D) and fully three-dimensional (3-D), were completed with a Newtonian constitutive equation. 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. Preliminary modeling results in a 2-D model that uses the highly shear-thinning Carreau fluid indicated that very low viscosity regions near walls and faster filling occur compared with the Newtonian results. Our simulation found that the fluid pools near the center of the channel instead of being evenly spread throughout the distributor, suggesting a possible distributor redesign. Important physical properties, such as thermal conductivity and heat capacity of the feedstock, were measured and will help us to determine the temperature of the moving front as the material fills the mold.

We performed a general full factorial design of experiments to model the effects of changes in feedstock composition and processing parameters on the rheological behavior of compounded material. The objective of this experiment was to minimize the viscosity of the feedstock and its variation. Experimental results showed: (1) increasing solid loading increased the viscosity of the feedstock, and the consistency of compounded material deteriorated, as was indicated by the increased variation in the viscosity; (2) increasing surfactant (stearic acid) content decreased the viscosity and improved the consistency of the ceramic suspension; and (3) at high solid loading, increasing mixing time decreased the viscosity of the compounded material. In addition, increasing mixing time also reduced the variation in viscosity regardless of the amount of solid loading.

We performed a fractional factorial experimental design to quantify the effects of injection molding parameters on the physical dimensional control of injection-molded parts. Parameters that we found to be statistically significant and could be set to achieve various dimensional targets were identified. We accomplished the data analysis after the dewaxing process and continued monitoring the changes in physical dimensions after the final sintering processes.

We performed an experiment to understand the defect formation during the injection molding process by varying the molding parameters. Preliminary results suggest that a combination of lower temperature (120 °C) and medium plunger speed (80%) tends to minimize defect formation and assure complete mold filling. Preliminary results from micro-computer tomography (μ-CT) analysis on injection molded ZnO parts suggest there are a few molding related defects in some injection molded samples. We are currently trying to establish a correlation between these defects and the processing parameters.

Experimental results indicated that stearic acid modified the lead zirconate titanate (PZT) surface and enhanced the dispersion of powder. Diffuse reflectance infrared (IR) spectra showed that chemisorption of the carboxylic acid group from stearic acid onto the PZT surface permits its long carbon chain to be extended into the organic carrier system, providing a steric stabilization that hinders the particles from exerting mutual attraction. Therefore, the formation of carboxylate compound enhances the particle dispersion in the organic system.

## Refereed

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## Other Communications

C.B. DiAntonio, P. Yang, G.R. Burns, and M.B. Nicholas, "Optimizing Organic Pyrolysis Profiles Using Master Decomposition Curves for Injection Molded Components" presented at the Rio Grande Regional Advanced Materials Symposium, Albuquerque, NM, October 2004.

R.R. Rao, T.A. Baer, L. Mondy, L. Halbleib, A. Grillet, and P. Yang, "Injection Molding of a Ceramic Suspension: Rheological Measurement and Computational Modeling," presented at the Society of Rheology 76th Annual Meeting, Lubbock, TX, February 2005.

P. Yang, C.B. DiAntonio, and G.R. Burns, "Injection Molding of Lead Containing Piezoelectric Ceramics," presented at the Center of Innovative Sintered Products Member Meeting, University Park, PA, November 2004.

L.L. Halbleib, P. Yang, L. Mondy, and G.R. Burns, "The Effects of Process Parameters on Injection-Molded PZT Ceramics Part Fabrication - Compounding Process Rheology," Sandia Report SAND2005-2864, Albuquerque, NM, 2005.

C.B. DiAntonio, P. Yang, and G.R. Burns, "Discovering Master Decomposition Curves for Organic Pyrolysis of Injection Molded Components," presented at the PIM 2005 International Conference on Injection Molding, San Diego, CA, March 2005.

R.R. Rao, T.A. Baer, L. Mondy, L.L. Halbleib, A. Grillet, and P. Yang, "Computational Modeling of Injection Molding of Ceramic Parts," presented at the International Workshop on Numerical Methods for Nonnewtonian Fluids, Santa Fe, NM, June 2005.

P. Yang, L.L. Halbleib, and G.R. Burns, "The Thermal Rheological Behavior of Compounded Lead Zirconate Titante Ceramic," presented at the PIM 2005 International Conference on Injection Molding, San Diego, CA, March 2005.

L.L. Halbleib, P. Yang, G.R. Burns, and M.B. Nicholas, "The Effects of Various Material Compositions on the Injection Molding Properties of PZT Ceramics," presented at the American Ceramic Society 107th Annual Meeting, Baltimore, MD, April 2005.

P. Yang, C.B. DiAntonio, L.L. Halbleib, D.R. Tallant, L. Mondy, and G.R. Burns, "Injection Molding of Lead Zirconate Titanate Ceramics," presented at the American Ceramic Society 107th Annual Meeting, Baltimore, MD, April 2005.

# Macro- Meso- Microsystems Integration in LTCC

*67008*

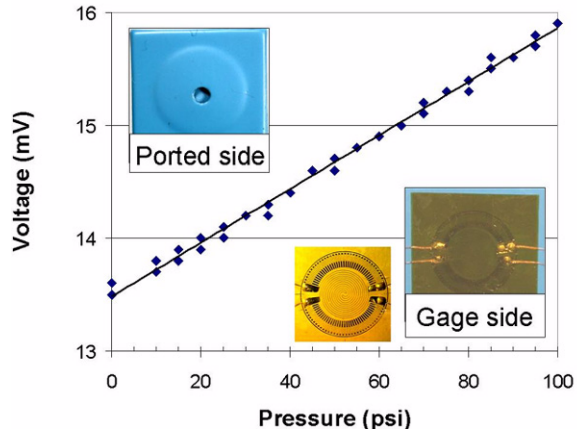
*K. A. Peterson, C. D. Nordquist, B. R. Rohrer, C. A. Walker, C. K. Ho, K. D. Patel, K. B. Pfeifer, A. L. Casias*

## Abstract

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

Routing of microelectronic traces through available space on any layer is a feature of most microelectronic board technologies. Our innovation using sacrificial volume material now makes this same routing versatility available for open volumes in multipurpose boards for microsystem integration. In addition to serving as microfluidic channels for dispensing, sensing, or cooling, these volumes can integrate microelectronic and microsystem devices, as well as provide surfaces for new sensing materials in smart channels. This can be accomplished with newly developed suspended structures that provide low electrical loss and low mass. The incorporation of metallic elements in the structure will permit higher integrity leads, heat sinks for superior heat removal, and electrostatically-actuated valves for microfluidic flow and pressure regulation.

We developed a number of innovative structures, whose properties are solving problems in real programs. In addition to capabilities in thin boards, bulk structures have been packed with a high density of microfluidic channels. We are refining newly developed capability for manufacturable, densely packed internal volumes, successfully integrating macro-, meso-, and micro- (MMM) scale features for systems. We are characterizing newly functionalized volumes, capable of electrokinetic pumping, temperature and pressure measurement, and actuation and switching. We will refine integral metal elements for structural, electrical, thermal, and fluidic features required in new applications. Many new structures developed by this team will be characterized and implemented in other Sandia projects. This work is expected to provide the first entry level modular approach to hybrid and multi-chip module microsystem integration. MMM system technology using LTCC will bring together microsystems and mesosystems and present them to a macroscale world.




*Pressure readouts from an LTCC diaphragm indicate a useful pressure sensor.*

## Accomplishments

The accomplishments for FY 2005 are complete or on track, and continue to provide significant improvements. While continuing the mesoscale demonstrations that have proven successful, we have also made progress on incorporation of bulk metals. We designed and fabricated a working cell lyser. We designed and fabricated micro-high performance liquid chromatography modules that even incorporate electrodes for electrokinetic pumping. We made innovative new use of sacrificial materials, including new methods and materials. The use of setter as sacrificial material has led to moving parts: spinning wheels, cantilevers, membranes, and plates for the first time in the world. Fabrication of both hybrid and cofired pressure sensors has been accomplished and is under further development. All previous attempts involved assembly instead of cofiring. Fabrication of thermal sensing structures on suspended thick films led to unprecedented sensitivity for this type of structure. Fabrication of a switch mechanism using suspended thick films is another first in the world. Fabrication of dense arrays of microfluidic channels has become more familiar to us and attracted

much interest from other programs. Fabrication of test manifolds for microfluidics will undoubtedly facilitate testing of a huge variety of devices arriving from microsystem designers. This includes high fluid flow devices for analysis of toxic materials. We fabricated a smart channel, which is performing well and is able to detect several solvents and substances at least as well as its non-LTCC predecessors. The work of the team resulted in a conference presentation and proceedings manuscript that was very well received internationally, an invitation for the principal investigator to participate as a panelist in a microelectromechanical systems packaging session at the INTER-Pack 05 packaging conference, and an invitation and acceptance for publication of a manuscript in a refereed journal. Although our own prior art likely prevents patenting of novel modular integration approaches, indications are that these will be well received in other programs.

## Other Communications

K.A. Peterson, K.D. Patel, C.K. Ho, S.B. Rohde, C.D. Nordquist, C.A. Walker, B.D. Wroblewski, and M. Okandan, “Novel Microsystem Applications with New Techniques in LTCC,” in *Proceedings of the 1st International Conference on Ceramic Interconnect and Ceramic Microsystems Technologies*, 2005, CD-ROM, pp. 156–173.

K.A. Peterson, K.D. Patel, C.K. Ho, S.B. Rohde, C.D. Nordquist, C.A. Walker, B.D. Wroblewski, and M. Okandan, “Novel Microsystem Applications with New Techniques in LTCC,” to be published in *Journal of Applied Ceramic Technology.*

K.D. Patel, K.W. Hukari, K.A. Peterson, “Low Temperature Cofired Ceramic Substrate Technology for Microfluidic Systems: Development of a High Temperature Ceramic Lyser for Solubilizing Spores,” in *Proceedings of the Conference on MicroScale BioSeparations*, 2005, CD-ROM.

# Design and Manufacture of Complex Precision Optics

*79741*

*D. D. Gill, B. L. Spletzer, M. P. Saavedra, M. J. Vasile*

## Abstract

The miniaturization of security and defense systems is creating a need for high precision micro and mesoscale optical systems. This is especially true in micro-robotics 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 that uses the Moore 350FG Freeform Generator and wire EDM (electro-discharge machining).

This research will investigate 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 optics' complexity is beyond the capability of current processes and will require the development of new process enhancements and integration of multiple manufacturing processes. These capabilities will enable Sandia designers to greatly increase the complexity of designs while reducing the overall size for both nuclear weapon (NW) and non-NW systems.

Research challenges will be defined by the development of a revolutionary micro-optical, multi-layer, fly-eye microlens array for micro-robotics. These microlens arrays will 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 charge-coupled device (CCD). Fabrication of the lenslets and registration, front-to-back, will be the greatest challenges.

Collaborative research will develop manufacturing and design technology for a multi-layer array of precision hybrid diffractive/refractive micro-optics, and precision alignment and mounting components produced by EDM.

## Accomplishments

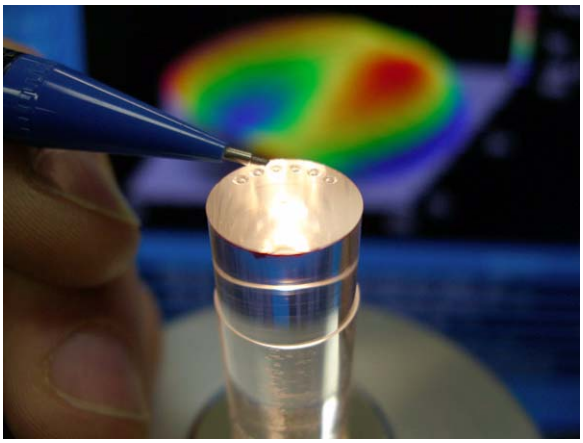
The goal of the project is to develop a new capability for creating complex optics within NW components. To develop this capability, we proposed to address the following three research and design focus areas:

(1) Design and manufacturing organizations must understand each other's capabilities such that optical designs optimize a balance between performance, manufacturability, and design expediency.

(2) Manufacturing Tooling: There are numerous innovative tools, methods, and fixtures that must be developed to provide rapid response to future customers' needs.

(3) A highly complex compound optical system, the Dragonfly Eye, would be fabricated as a demonstration test bed.

Our accomplishments toward these three goals have been significant. We created multiple design iterations for the proposed Dragonfly Eye and, through that process, the team has developed an understanding of how to translate optical tolerances into manufacturing tolerances and how to design for the manufacturing capabilities. We filed a technical advance for the concept.



*These plunge-milled optics have a 600 µm diameter and are similar to those that will be arrayed around the outside of the acrylic Dragonfly Eye optical shell.*

One challenge in creating the Dragonfly Eye array is the creation of 80 mesoscale, aspheric lenslets on the exterior of the dome. To answer the challenge, the team created an innovative means of manufacturing concave, aspheric, diamond mills. This revolutionary tech-

nique is required anytime an optically-corrected lenslet needs to be created off-axis, as in arrays of lenslets. The diamond mill is being modified by Focused Ion Beam (FIB) to create a precision asphere. The project team has thus far experimented with modification of a tool, FIB/diamond erosion rates, and generation of path planning for the modification process. Outside entities have queried the team about this capability, and excitement over this technique is building.

Finally, we created test arrays of mesoscale optics in acrylic using both milling and turning techniques. These arrays are being evaluated to determine the transfer function between tool shape and the shape of the optic produced. This knowledge will be utilized in the manufacture of the aspheric mills to produce optics with the highest possible accuracy.

Specific accomplishments of the project team include:

- Design iterations completed for multi-layer, complex, mesoscale, Dragonfly Eye optic. Design includes manufacturability as a consideration to ensure success.
- Arrays of meso-scale optics (600 µm diameter) have been created by both diamond milling and diamond turning and are being evaluated to determine accuracy.
- FIB machining of diamond has determined erosion rates and initial methods of path planning for diamond tool modification. Outside entities (potential future customers) are showing a great interest in this capability.
- Research has produced two paper presentations.
- Research has inspired three new customers to fund projects requiring optical design and manufacture.

## Refereed

W.C. Sweatt, D.D. Gill, D.P. Adams, M.J. Vasile, and A.A. Claudet, “Diamond Milling of Micro-Optics,” in *Proceedings of the IEEE Aerospace Conference*, 2006, CD-ROM.

## Other Communications

W.C. Sweatt, D.D. Gill, G.A. Vawter, and J.D. Williams, “New Fabrication Techniques Enable New Optics Designs,” presented at the Annual Meeting of the Optical Society of America, Tucson, AZ, October 2005.

# Development of a Manufacturing Capability for Production of Ceramic Laser Materials

*79742*

*J. A. Voigt, P. Yang, T. J. Garino, S. J. Lockwood, J. Cesarano III, R. L. Schmitt, L. S. Weichman, C. C. Phifer*

## Abstract

Optical engineering technology, such as direct optical initiation (DOI), is a key to future advanced firing/detonation and other weapon subsystems. A critical component of the DOI firing set architecture is a solid-state laser that relies on a unique, radiation hard Nd-doped gadolinium scandium gallium garnet (GSGG) single crystal material. A sole-source supplier using an expensive, inherently problematic and unreliable Czochralski method grows the GSGG single crystals on an as-needed basis. The ability to replace the GSGG single crystal with the ceramic counterpart will not only eliminate these problems, it would also provide many other cutting edge opportunities. We propose to develop an in-house production capability to prepare GSGG and other laser materials in ceramic form. Although, to date no transparent, laser-quality ceramic GSGG has been reported, the Japanese have commercialized laser-quality ceramic Nd-doped yttrium aluminum garnet (Nd:YAG) that is not radiation hard. Building on the work of the Japanese, we will use our extensive experience to develop a GSGG production process by integrating (1) solution synthesis of ceramic powders, (2) green body formation and sintering science, and (3) process scale-up. Further, by coupling this new process with Sandia's direct-fabrication technologies (e.g., robocasting), we will develop the ability to do graded doping and to build extended structures. These processing technologies will make rapid prototyping of new optical ceramic materials for advanced weapon subsystems possible.

## Accomplishments

The progress to date in the first year of this project has been substantial and is achieving stated milestones. Research efforts focused on development of a chemical solution route to produce powders for the YAG ceramic laser material system. We generated enough results to present a paper on our work at the Annual Meeting of the American Ceramic Society, Baltimore, April 10–13, 2005. It is worth noting that ceramic laser materials development was of high interest at the meeting, as evidenced by the large number of talks devoted to it. In addition to the Japanese continuing to have a strong presence in the field, significant efforts are under way at Pennsylvania State, Stanford University, and Lawrence Livermore National Laboratory.

We initiated our work this year by completing an in depth literature review of YAG powder synthesis and processing. The summary of the review provided a step-by-step analysis of each of the synthesis and processing steps, and general guidance for each with respect to the choice of the range of experimental parameters. For example, temperature ranges and times for the calcination of precursor powders to the crystalline garnet phase were generated. Based on the literature review, and our own experience in developing solution routes to ceramic powders, hydroxycarbonate and oxalate precipitation routes were chosen for investigation. Initial results at the small batch-scale showed that the hydroxycarbonate route produced finer, more sinterable powders. Since there were no obvious advantages with regard to filterability for the oxalate-based system, as is often seen, the hydroxycarbonate route was chosen for further development.

One of the keys to producing the essentially pore-free microstructure required by the laser application is the preparation of highly uniform green bodies from the calcined powder. To achieve this, a powder generally must be conditioned by processes such as milling or sonication, and then formed into the green body by dry pressing, slip casting, or some other consolidation process. The calcined YAG powder prepared by the process described above consists of agglomerates composed of approximately 100 nm primary crystallites. To facilitate uniform compaction, we investigated a variety of combinations of Spex milling, ball milling, and high intensity ultrasonic disruption. In Spex milling, a powder or powder slurry is placed in a canister made of a hard material (we used either zirconia or tungsten carbide) along with a bead of the same material. The closed assembly is then shaken at a very high intensity to break up the powder. Using these approaches, break-up processing parameters were established to reduce the mean agglomerate size from several microns to below 500 nm.

We also extensively investigated compact formation. Calcined powders were either processed as milled or as dispersions with Darvan C being used as the dispersant. We also investigated the use of the binders polyvinyl alcohol (PVA) and polyethylene glycol (PEG) to improve powder flow properties and compact uniformity. In addition to the previously mentioned dry pressing and slip casting, we also used compact formation by vacuum and centrifugal casting methods. The scanning electron microscope (SEM) photomicrographs illustrated both the fine morphology of the chem-prep YAG powder and the uniformity of a slurry-processed compact. The compacts were bisque fired at up to 1000 ºC to remove any residual organics that were incorporated during the powder-conditioning step. In order to prevent the capture of gases during the densification process, sintering was generally performed in vacuum (< 1 mT) at temperatures from 1600 to 1750 ºC for 5 hours.

To verify the activity of the chem-prep YAG powder's reactivity toward densification, dilatometry run in air (heating rate = 10 ºC/min.) showed that 99% of theoretical density could be achieved by 1650 ºC. Using the vacuum sintering process, we have been reasonably successful in producing samples with low residual porosity. Although we have prepared samples with a reasonable degree of transparency, they are not yet of laser quality.

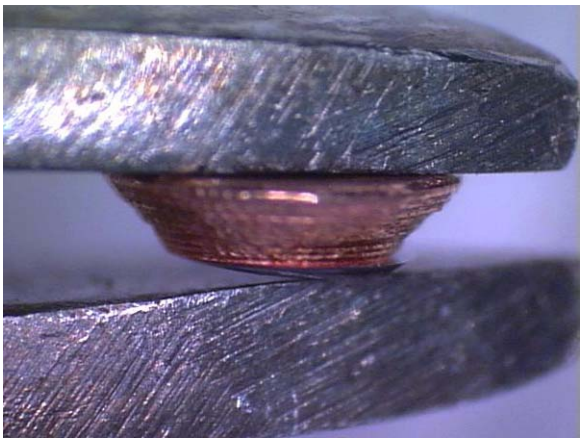
# Direct Writing in PMMA Photoresist to Expose Free-Form Geometries

*79743*

*J. A. Palmer, J. D. Williams, P. Yang, T. Lemp*

## Abstract

Deep x-ray lithography is currently used to define prismatic shapes in polymethylmethacrylate (PMMA). The x-rays lower the molecular weight of the exposed material, making it susceptible to etching. Similarly, a femtosecond laser can expose PMMA in the region of its focal point. Unlike other lasers, femtosecond lasers can easily create damaged volumes in the interior of transparent materials including PMMA, without damaging the surface. We have shown that this laser-damaged volume is affected by conventional LIGA (for the German term *Lithographie*, *Galvanoformung*, *und Abformung*, for lithography, electroforming, and molding) developer. This maskless process can define complex non-prismatic geometries, one layer at a time, by writing lines (overlapping lines if necessary) starting at the bottom level in the PMMA and working upward. Also, PMMA that has been x-ray exposed can then be laser exposed to create additional geometry. For example, the laser can define multilayered helical coils around an x-ray defined mass. Or, laser defined fasteners can be inserted, joining x-ray defined parts. Overhang can be added to an existing part. Electrical interconnects and vias can be laser defined. By using the laser to damage the PMMA and not to ablate it, no debris is created and high resolution geometries can be defined. The PMMA, whether x-ray exposed or laser exposed, may be developed and plated using conventional LIGA processes. We do not expect the laser-defined geometries to be as precise as the x-ray defined geometries. We expect the laser spot size to be on the order of 5 microns and the positional stages to be accurate to within 2 microns. This project is proposed to identify ideal parameters for near infrared femtosecond laser processing of PMMA complement LIGA with directly written laser features to realize the following mesostructures: (1) a three-dimensional (3-D) conical washer or bevel gear created by laser augmentation of existing two-dimensional features, and (2) a 3-D integrated micro-fluidic connector for use in heat pipes and numerous micro fluidic devices.



*3-D copper LIGA component.*

## Accomplishments

(1) We designed a fractional factorial experimental matrix to identify the optimal parameter set for laser processing of PMMA. We studied four parameters including laser fluence, substrate feed rate, polarization rotation speed, and developer solution temperature. We drilled an array of 500 µm diameter holes through the sample to mimic pre-existing features. The laser beam was focused and scanned within the bulk at each hole site to define an offset cylinder, 500 µm in diameter and 150 µm high. Experiments were run in ambient atmospheric conditions. The beam was scanned "bottom to top," moving upward toward the PMMA surface in 20 µm vertical steps. The overall position of laser-processed cylinders was varied, i.e., some were buried and some were created at or near the surface. Following laser processing, we immersed the PMMA sample in LIGA developer solution for four hours. We subsequently sectioned and examined the laser-defined cylinders under an optical microscope. The optimal parameter set minimized an empirical response function. The study identified the following optimal parameters: (1) fluence at 0.8 J/cm^2 (high), (2) feed rate at 200 µm/s (high), (3) polarization rotation speed of 60 rpm (low), and (4) development temperature at 40 ºC (high).

(2) We recorded optical micrographs (top view and cross section) of 150 µm cubic voids created using

optimal parameters within the bulk and at the surface of 1 mm thick PMMA substrates. We conducted a side-by-side comparison of identical buried cubes. One was immersed in the developer solution while the other was not. Although a change in appearance was evident, the developer solution did not evacuate laser exposed material according to the hypothesis.

(3) We designed a 3-D (800 µm diameter, 200 µm high) cone based on a simple cylinder. We chose the cone over the original annular augmented LIGA part design to facilitate plating. We wrote a program to define the part in bulk PMMA by scanning the laser beam in an alternating xy pattern. The complete part geometry was successfully defined by ablation. However, ablation damage to the metal seed layer at the base of the part caused failure of the plating step.

(4) We pursued two contingency processes. The first involved frequency doubling the nominal femtosecond laser system and processing PMMA samples that were doped with ultraviolet absorbers. Optical micrographs of the doped sample cross sections are currently under review. The second employed laser-assisted micro stereolithography to add 3-D micro molds to existing LIGA features. This contingency was successfully implemented and yielded the 3-D part described above. We submitted a technical advance.

## Refereed

J.A. Palmer, J.D. Williams, T. Lemp, F. Medina, and R.B. Wicker, “Advancing Three-Dimensional MEMS by Complementary Laser Micromanufacturing,” to be published in *Proceedings of the SPIE Photonics West*, 2006.

# High Speed, Extended Depth of Field, Multi-Spectral, 3-D Imaging System for Real-Time Micro Assembly of MEMS

*79744*

*J. J. Carlson, B. A. Kast, C. W. Wilson, D. K. Novick*

## Abstract

Imaging systems for microelectromechanical systems (MEMS) inspection and assembly suffer from several limitations. Depth of field is extremely narrow at large magnifications. Many inspection and most automated assembly processes require real-time three-dimensional (3-D) information regarding the position, orientation, and shape of components. When component dimensions are less than the wavelength of light, traditional imaging is not even possible. We propose to develop a revolutionary, high-speed, extended depth of field, multi-spectral, 3-D imaging system useful for MEMS inspection and assembly operations using a narrow band, liquid crystal, tunable filter. The system will enable rapid, accurate inspection of MEMS components and real-time control of micro-manipulation systems used for MEMS assembly.

The basic concept uses a white light source for illumination and a high-speed, liquid crystal, tunable filter to exploit the chromatic aberrations characteristic of simple lenses. The MEMS components will be imaged using the electronically tunable filter across several narrow spectral bands. Component details imaged at a specific distance from the lens will appear in sharp focus only in one spectral band. Details at other distances will appear out of focus in the same band. By imaging components across several spectral bands and combining the images using specially developed algorithms, a composite, extra sharp focus, extended depth of field, multi-spectral, 3-D image can be obtained. The proposed developments are unique in that the imaging system does not employ any mechanically driven stages. It is vibration free and fast, both critical requirements for precision, large scale micro assembly operations. These unique capabilities will enable autonomous, high-speed MEMS inspection and assembly processes that are currently unavailable from other imaging technologies.

## Accomplishments

The goal was to develop a high-speed, extended depth of field, multi-spectral, 3-D imaging capability that could be used to develop a semiautonomous micro assembly work cell. The sensor could be used to autonomously control a precision manipulator used in a micro assembly process. A major accomplishment was the successful development and demonstration of an optical system that could acquire a multi-spectral focus series without the need for any mechanically driven elements or stages. We anticipated using existing algorithms for producing extended depth of field images and 3-D surface profiles by processing a focus series. Although many existing algorithms we evaluated were useful for producing an extended depth of field image from the focus series, none were adequate for producing 3-D surface profiles. The reason is that, at low magnifications (< 10), there are regions across images of typical components that have little or no texture. Under this condition, it is impossible to extract 3-D information from a focus series. We believe, however, that this problem will rapidly diminish in applications requiring much greater magnification (at high magnification, images of even relatively uniform objects appear with significant texture).

We also evaluated commercial systems that were designed to operate from a focus series. All of these could produce very good extended depth of field images but none were adequate for 3-D surface profiling. These systems create an extended depth of field image from a focus series obtained by mechanically moving the entire imaging system up and down. Because the algorithms used in these previous systems proved inadequate, we were unable to achieve our original goals. We spent a significant, unanticipated effort trying to improve on existing algorithms. Our initial algorithm developments were extremely successful in that they significantly outperformed existing algorithms. We believe it is possible to obtain even better results with new algorithms that maximally exploit the chromatic aberrations characteristic of simple lenses used in our approach. The detection and measurement of these aberrations form the basis for the entire approach.

# Optics Integration into Microsystems Using Electrostatically Steerable Fibers

*84273*

*K. A. Peterson, O. B. Spahn, B. Jokiel Jr., B. Wroblewski*

## Abstract

Integration of optics is essential to microsystems. Microelectromechanical systems (MEMS) optical switches are receiving special attention for their use in telecom, space, and medical functions. These functions are typically based on MEMS mirrors/shutters and free-space coupling, which can involve large distances. Although the MEMS elements are small, the necessary infrastructure is not. Practicalities require increased spacing to mitigate interference, area for comb drives or other actuators, and packaging involving high voltages for actuation. Even with the required practicalities accomplished, MEMS mirrors cannot handle large amounts of power, and they need to be coupled to fibers at the subsystem perimeter.

Several researchers have sought enhanced capability by aligning fibers directly in a switch. Some build elaborate MEMS devices, only to use them as crude bumpers to nudge fibers into place. They depend on the same alignment principles that allow manual gluing of aligned fibers (no better than machined grooves). Others build large bulky mechanisms that do align fibers electrostatically, but at a considerable size premium.

We will develop microsystem boards and packages that steer integrated signal fibers to their mates electrostatically through a large range of available positions. This can be used for switching or alignment. The technique can take advantage of voltages already supplied to comparable switches. Bearing surfaces are eliminated, thermal problems disappear, and free-space coupling distances can be exceedingly small. The electrostatic signal can be varied to optimize maximum power, making latching mechanisms possible by taking advantage of tip motion or motion gradients along the length of the fiber.

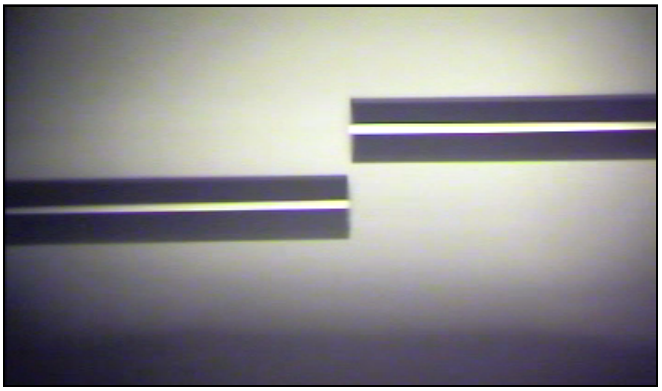
This work will result in a new capability for improving the alignment of optical fibers in critical applications, and will have considered the necessary manufacturing technology along the way in order to apply the benefits to Sandia programs.

## Accomplishments

We completed the milestones of this project. We completed a literature survey and have a clear understanding of prior work. We examined recent work in optical fiber mating for high performance, including review of theoretical behavior of cantilevered, electrostatically-actuated fibers as affected by material and geometrical parameters. We analytically examined the theoretical performance of single mode and multimode optical fiber with respect to effects of misalignment on coupling efficiency. We were able to investigate prototypes for “smart fibers” (electrodes on the fiber itself), a simple mechanical test fixture, and some prototype low-temperature co-fired ceramic (LTCC) fixtures. Several prototypes have been fabricated concentrating on a method of manufacture based on LTCC. These include fiber arrays that place multiple parallel fibers on a single board. This work was ahead of schedule, but needs additional development to demonstrate function and manufacturability.

We filed a patent application that highlights the unique advantages of our LTCC method of manufacture.

The final report for this project includes: (1) a slow motion demonstration of the capability to electrostatically steer and couple fibers while optically energized or not, and (2) a demonstration of the range of fiber diameters achieved by electrostatically steering an optically energized fiber with less-than-perfect prototype electrodes.



*Movie demo of steering and signal coupling.*

# Biotechnology

## Modeling Biomembranes

*52532*

*M. J. Stevens, A. L. Frischknecht, S. J. Plimpton, D. Y. Sasaki, L. J. Frink*

### Abstract

Understanding the properties and behavior of biomembranes is fundamental to many biological processes and technologies. Microdomains in biomembranes, or "lipid rafts," are now known to be an integral part of cell signaling, vesicle formation, fusion processes, protein trafficking, and viral and toxin infection processes. Understanding how microdomains form, how they depend on membrane constituents, and how they act, not only has biological implications, but will also impact Sandia's ability to develop membranes that structurally adapt to their environment in a controlled manner. To achieve such an understanding, we propose to create physically based models of biomembranes.

Using these models, we will apply molecular dynamics (MD) simulations and classical density functional theory (DFT) to phenomena such as microdomain formation, membrane fusion, pattern formation, and protein insertion. Due to lipid dynamics and self-organization in membranes occurring on length and time scales beyond atomistic MD, we will use coarse grained models of double tail lipid molecules that spontaneously self-assemble into bilayers. Using such a model, we already demonstrated that the time scales of lipid diffusion and fusion intermediates are accessible to MD simulations. This model enables the study of a whole set of membrane dynamics including those listed above.

These coarse grained models can also be used in our DFT code. DFT will provide equilibrium information on membrane structure; free energies and routes to assembly of membrane bound proteins; and free energies of permeation for water, ions, proteins, and toxins as they traverse the membrane. We will perform experimental work to help elucidate the fundamental organization principles. In summary, this project will produce significant new understanding of biomembrane structure and dynamics and build a strong foundation of physically based models for further development.

### Accomplishments

We performed a variety of calculations both to broaden our understanding of coarse grained model lipids and to make direct contact with experiments. Model development addressed the two main membrane problems of interest: domain dynamics and protein lipid interactions. We specifically examined phosphatidylcholine lipid mixtures to more fully understand the range of domain behavior for a simple system. We used our coarse grained models to study important aspects of protein-lipid interactions not possible with other models (i.e., atomistic models). In particular, we studied the dynamics of peptide insertion into a lipid bilayer. We compared results of classical DFT to MD simulations of coarse grained models of lipids in solvent. We found that the DFT adequately captures the liquid structure of coarse grained lipids both near surfaces and in bilayers. The mechanical properties of the fluid DFT bilayers are qualitatively similar to the MD bilayers. However, at low temperatures we found that in the MD simulations, the ordering in bilayers is different from the DFT calculations.

### Refereed

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Bilayers," to be published in *Physical Review E*.

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# Reverse Engineering Biological Networks: Applications in Immune Responses to Bioterrorism Threats

*52533*

*J. M. Faulon, S. B. Martin, A. Slepoy, J. A. Timlin, D. M. Haaland, G. S. Davidson, A. Martino, Z. Zhang*

## Abstract

Our goal is to determine the network of events, or the regulatory network, that defines an immune response to a biotoxin. We anticipate that by inferring a regulatory network, we will be able to isolate a handful of specific regulatory elements that fingerprint a response. Ultimately, this work will provide the blueprint of a simple experiment where the biotoxin is characterized by measuring only the fingerprint elements rather than the entire immune response. This goal addresses the homeland security mission of the Department of Energy (DOE). Regulatory network characterization is also an explicit goal of the Genomes to Life (GTL) DOE program.

As a model system, we are studying a T cell regulatory network triggered through tyrosine kinase receptor activation, using a combination of pathway stimulation and time series microarray experiments. Our approach is composed of five steps: (1) microarray experiments, (2) data error analysis, (3) data clustering, (4) reverse engineering, and (5) network dynamics analysis and fingerprint identification. The technological outcome of this study will be a suite of experimental protocols and computational tools that are able to reverse engineer regulatory networks from a minimum amount of data. The practical biological outcome of this work should be the discovery of immune response fingerprints to a biotoxin in terms of expression levels of regulatory elements.

Inferring regulatory networks from microarray data is a new field of investigation that is no more than five years old. To the best of our knowledge, this work is the first attempt that integrates experiments, error analysis, data clustering, inference, and network analysis to solve a practical problem. While microarrays have been used with time series experiments and gene or pathway stimulation experiments, we are not aware of any study merging both types of experiments. The systematic approach of counting, enumeration, and sampling is new to the field of network reverse engineering. The resulting mathematical analyses and computational tools should lead to new results on their own and should be useful to others who analyze and infer networks.

## Accomplishments

T cells first respond to biotoxins by producing interleukin-2 (IL-2), a protein that triggers a complex immune response, leading T cells to proliferate and differentiate. In FY 2005, we characterized this immune response by determining a dynamical fingerprint in gene regulation. This fingerprint is composed of four classes.

(1) Early response genes (up-regulated after 1 hour): This category of genes, such as irf4 and Bcl2, prevents programmed cell death. This is expected, as T cells stimulated by IL-2 are not supposed to die but instead proliferate and differentiate.

(2) Cyclic response genes (up-regulated after 1 hour and again after 16 hours): These genes regulate cell cycle and should indeed have cyclic expression profiles. This is additional evidence that IL-2 stimulated T cells are alive.

(3) Intermediate response genes (up-regulated after 4 hours): In this class, we find genes such as Rpo1, Hnr, and Bcl3 that are involved in transcription and, thus, induce protein production.

(4) Late response genes (up-regulated after 8 hours): This class comprises genes such as Mybl2, Foxm1, and Nsbp1 that are regulators (transcription factors) of other genes. Up-regulation of transcription factors indicates the message sent by IL-2 stimulation was taken into account and the immune response was triggered.

To obtain the above results, we cultured IL-2 dependent T cells to mid-log phase (~2 x 105 cells/ml), collected them by centrifugation, and grew them in D10 medium with no IL-2 (IL-2 starvation) for 14 hours. We collected cells (0 hour, no IL-2 stimulation) immediately before we added IL-2. We also collected cells at series time points after IL-2 stimulation at 15 minutes, 30 minutes, 1 hour, 2 hours, 4 hours, 6 hours, 8 hours, 10 hours, 12 hours, 16 hours, and 24 hours. We isolated total ribonucleic acid (RNA) (transcripts of all the genes) and used it to synthesize biotin labeled total complementary RNA (cRNA) and hybridized onto Affymetrix GeneChip Mouse Genome 430 2.0 Array (45,000 probe sets with over 34,000 well characterized mouse genes). After 16 hours of hybridization, we

washed the GeneChips and scanned them using the Affymetrix system to generate gene expression data.

We compared each gene from series time points to that of time point 0 hour to determine its expression level and expression trend (up-regulated or down-regulated) in response to IL-2 stimulation. We found that 5890 genes differentially expressed above 1.5 fold. We next clustered the 5890 genes, using a k-mean algorithm, into 23 clusters falling into four categories: early, cyclic, intermediate, and late response genes, as described above. The averaged expression profiles of the clusters were fed to a gene regulatory network inference algorithm (GENET) developed in the two previous fiscal years. Several possible networks were returned by GENET. Out of the solution set, one network exhibited higher stability and robustness. This network produced the same dynamics after perturbations of reaction rates and initial gene expressions. Furthermore, at steady state, we found this network to have a cyclic pattern in gene expression ranging between 16 and 24 hours. This finding is consistent with known cell cycle lengths for typical mammalian cells.

Additionally, in conjunction with other LDRD and GTL projects, we enhanced our hyperspectral image analysis methods and software (used in this project to scan microarray chips). Thus, we generated upgraded C-based multivariate curve resolution software (AXSIA-Automated eXpert Spectral Imaging Analysis) that extracts pure component emission spectra and relative concentration maps from hyperspectral image data. The AXSIA software was upgraded with equality constraints, selective non-negativity constraints, input of initial spectral starting points, and spectral residual analysis. All these new capabilities are required for rapid analysis of hyperspectral fluorescence images.

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# High Throughput Identification of Molecular Machines Involved in Membrane Signaling and Toxin Pathways

*52536*

*A. K. Singh, S. P. Gaucher, R. Sapra, M. Z. Hadi, S. Chhabra*

## Abstract

The purpose of this project is to identify proteins involved in host-pathogen response for interaction of anthrax toxin with human and mouse macrophages. These proteins can serve as potential biomarkers to develop next generation diagnostic assays and can also be used as targets for therapeutic agents.

*Bacillus anthracis*, which is the etiological agent for anthrax, recently gained wide coverage in the news media for its use as a bioterrorism weapon. The bacterium produces and secretes anthrax toxin (AT), which is comprised of three polypeptides: protective antigen (PA), lethal factor (LF), and edema factor (EF). Although there are three proteins that make up the whole toxin, AT is, in fact, a binary A-B toxin where a combination of PA (component A) and either LF or EF (component B) can infect and cause cell damage. A combination of PA and LF is commonly referred to as the lethal toxin (LT). The same combination with PA and EF is referred to as the edema toxin (ET), with PA being the important factor for infection in both combinations. We are currently using LT and ET in separate experiments to understand the mechanism of action and the pathways affected by the toxin. We are also working to understand the biomarkers expressed by a cell in response to the infection that may help in developing diagnostics for biodefense.

## Accomplishments

Murine macrophage (mMP) cell lines and human dendritic cells (hDCs) were selected as hosts to be exposed to the lethal toxin for 2 to 24 hours. The resulting cell death is monitored using a cell counting chamber to obtain a time plot of cellular apoptosis. The plot is then used to generate the exact experimental conditions for comparison between the control population and the toxin infected population. Based on the results of the apoptosis analysis, the cell samples collected for analysis are lysed, precipitated, and then resuspended in denaturing buffer; and the samples are prepared for separation and analysis by two-dimensional (2-D) gel electrophoresis.

After the initial analysis and protocol development, we covalently linked the protein samples to cyanine dyes and prepared them for 2-D differential in gel electrophoresis analysis (DIGE). We scanned the resulting 2-D gels and analyzed them for changes in protein expression. We excised the regulated protein “spots” from the gel using a robotic spot picker and deposited them into 96 well plates. We then subjected the gel plugs containing proteins of interest to proteolysis using trypsin. We collected and analyzed the resulting peptides by matrix assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectrometry.

We exposed the mMP and hDC to a combination of PA and LF and monitored the cell death every 30 minutes. Analysis of cell death shows that the mMP cells start lysing after 55 minutes of exposure and are completely (> 95%) lysed after 120 minutes of exposure to the toxin. The hDCs, on the other hand, do not undergo cell lysis even after > 24 hours of exposure to the toxin. These observations are consistent with results reported in the literature.

We collected, lysed, and prepared the infected and control mouse macrophage cells for 2-D analysis, as described above. Preliminary analysis of the control cell proteome shows that there are over 3500 proteins that can be visualized using the 2-D gels over a broad pH range. We categorized the identified proteins into four major categories: proteins involved in energy production, cytoskeletal proteins, stress response proteins, and miscellaneous other proteins that are either of unknown function or have a broad range of functions. When J774A.1 mMP cells are challenged with LT, the electron transfer respiratory chain proteins, NADH dehydrogenase Fe-S protein and ubiquinol-cytochrome-c reductase complex core protein-I, which are components of the mitochondrial membranes, are up-regulated 2.7 and 2.3 fold, respectively. The other two proteins identified in energy production are ATPase subunits, also from the mitochondrial membranes. The cytoskeleton proteins beta-actin and A-X actin are up-regulated by 3.0 and 2.3 fold, respectively, and the nuclear membrane component lamin A is up-regulated 2.8 fold when the cells are challenged with LT. Stress

response proteins like heat shock protein (HSP) 70 and vimentin are expressed more than 2.1 fold under the same conditions. There are, however, five proteins that are regulated between 2.0–2.7 fold and have no characterized annotated function. Based on sequence analysis, these match the proteins involved in stress response, cytoskeleton, ribonucleic acid binding, and guanosine triphosphate binding proteins. When mMP-like RAW264.7 cells are challenged with LT, metabolic proteins like glyceraldehyde-3-phosphate dehydrogenase, dihydrolipoamide dehydrogenase, and enolase1 alpha are seen to be up-regulated between 2.2 and 2.3 fold. The ATPase β-subunit increased 2.7 fold as compared to LT cells. We found only one cytoskeleton protein, beta-actin, to be up-regulated in RAW264.7 cells. We identified more stress response proteins from RAW247.7: HSP 70, vimentin, glucose regulated protein (GRP) 78, and protein disulfide isomerase (PDI). These were consistently expressed in higher ratios as compared to the J774A.1 cells. Furthermore, the unclassified proteins identified have putative roles in oxidative stress response, in protein biosynthesis, and as chaperones.

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

*67010*
*A. R. Burns*

## Abstract

Cellular membranes have complex lipid and protein structures that are laterally organized for optimal molecular recognition and signal transduction processes. Knowledge of nanometer scale lateral organization and its function is important in the analysis of receptor based signaling, particularly signaling in response to toxins. We will use model membranes to perform a detailed study of the chemical and physical factors that result in lateral organization of lipids and lipid mediated protein sequestration into signaling domains. We will 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 Health Sciences Center. Our proposed development of new imaging methods that will provide both high spatial resolution and sensitivity to dynamical processes will enable our studies.

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. So far, 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 also propose to develop a fluorescence imaging technique that utilizes the highly localized excitation provided by the strong near field enhancement at a metal coated AFM tip. Overall, our unique imaging capabilities will enable us to examine membrane structure and function with much greater detail than previously possible and, thus, provide a better understanding of cellular signaling.

## 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 multi-component model membranes, we characterized partitioning of four fluorescence probes (headgroup and tailgroup labeled GM1 ganglioside probes and headgroup and tailgroup labeled phospholipid probes) between gel (saturated DPPC) and disordered fluid (unsaturated DOPC) phospholipid domains. The domains were mapped with the AFM topography. We 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.

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# Imaging Self-Organization of Proteins in Membranes by Photocatalytic Nanotagging

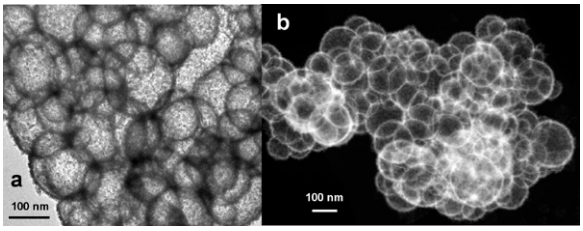
*67011*

*J. A. Shelnutt, C. J. Medforth, A. K. Singh, K. R. Zavadil, D. Y. Sasaki*


## Abstract

We developed a new nanotagging technology for detecting and imaging the self-organization of proteins and other components of membranes, at nanometer resolution, for the purpose of investigating cell signaling and other membrane mediated biological processes. Protein-, lipid-, or drug-bound porphyrin photocatalysts have been used to grow in situ nanometer sized metal particles, which reveal the location of the porphyrin labeled molecules by electron microscopy. Initially, photocatalytic nanotagging was used to image assembled multi-component proteins and to monitor the distribution of lipids and porphyrin labels in liposomes. For example, by exchanging the heme molecules in hemoproteins with a photocatalytic tin porphyrin, a nanoparticle can be grown at each heme site of the protein. The result obtained from electron microscopy, for a tagged multi-subunit protein such as hemoglobin, is a symmetric constellation of a specific number of nanoparticle tags—four in the case of the hemoglobin tetramer. We also developed general methods for covalently linking photocatalytic porphyrin labels to lipids and proteins to detect and image the self-organization of lipids, protein-protein super complexes, and membrane-protein complexes. We developed procedures for making photocatalytic porphyrin-drug, porphyrin-lipid, and porphyrin-protein hybrids for non-porphyrin binding proteins and membrane components, and investigated the first porphyrin labeled lipids in liposomal membrane models.

Molecular simulations can be used to determine label locations on proteins (e.g., the sites of formation of amide or sulfonamide linkages to lysines) and to model self-organization and cell signaling processes. A porphyrin-aspirin or another non-steroidal anti-inflammatory drug hybrid could be used to develop metal nanotags for the pharmacologically important cyclooxygenase enzymes in membranes so that the distribution of the protein can be imaged at the nanometer scale. Our photocatalytic nanotagging technique may ultimately allow membrane self-organization and cell signaling processes to be imaged in living cells. Fluorescence and plasmonic spectra of the tagged proteins could also provide additional information about protein association and membrane organization.


(a) *High resolution transmission electron microscopy (TEM) image of liposomes nanotagged with individual platinum "nano-daisies" that are linked to form a network that supports the spherical structure of the liposomes.* (b) *High angle annular dark field scanning TEM image of the platinum nanotag networks composed of joined dendritic nanosheets (nano-daisies).*

## Accomplishments

Building on early results with tin-porphyrin reconstituted hemeproteins, we generalized our photocatalytic nanotagging technique by synthesizing tin-porphyrin labeled lipids, making the nanotagging method applicable to systems that do not have native heme subunits that can be reconstituted with photocatalytic tin porphyrins. We investigated two general approaches and compared them with respect to labeling the lipid bilayers of model membrane systems: (1) incorporation of hydrophobic porphyrins within the bilayer of model lipid systems (where the nanotags are expected to grow between the layers), and (2) the design and synthesis of conjugates composed of a hydrophilic synthetic porphyrin headgroup, attached to various synthetic or natural lipids (where the nanotags are, thus, expected to grow at the porphyrin on the outer surface of the lipid bilayer). The porphyrin-lipid conjugates can be modified to adjust both the degree of hydrophilicity and the charge (positive or negative) of the porphyrin headgroup. This variability allows the labeled lipids to be incorporated into a range of membrane structures and also permits some degree of control over porphyrin-protein interactions.

We focused our efforts on understanding the distribution of photocatalytic porphyrin labels in liposomes, and in nanotagging particular lipids contained in liposomes. Our transmission electron microscopy studies showed that the location of the porphyrin photocatalyst, whether inside or outside of the membrane, can be distinguished by the type of platinum dendrite that grows at the platinum nanoparticle tag. When the porphyrin resides inside the membrane bilayer, the nanotag will grow into a dendritic sheet; when the porphyrin resides on the outer surfaces, the platinum nanotag will grow as a globular platinum dendrite. We also determined procedures for the direct attachment of hydrophilic synthetic porphyrins to proteins and synthesized the activated porphyrins required for this procedure. With this progress, we are confident that interactions of membrane proteins with liposomes and natural membranes are eminently feasible upon further development of this new nanotagging technique. Because the photocatalytically grown metal nanotags can be 1–3 nm in diameter, this nanotagging method offers enhanced resolution and the potential for in vivo application.

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# Protein Microarrays for Biowarfare Agent Detection and Characterization

*67012*

*A. E. Herr, A. K. Singh, D. M. Haaland, D. C. Roe*

## Abstract

We propose to leverage Sandia's strengths in microfluidics, membrane-protein expression, computational analysis of ligand binding, and hyperspectral microarray imaging to develop protein microarrays ("protein chips"). Technological means to quickly perform large scale functional analysis are crucial to closing the information gap between genomics and proteomics. Our proposed microarray concept mimics the molecular machinery of the host cell by arraying proteins, ligands, antibodies, and possibly phospholipids, sugars, and oligonucleotides. Protein chips promise to make a revolutionary advance in pathogen detection by enabling high-throughput, rapid (minutes or less) detection of a broad range of dilute (pM) threat agents. The system will perform a quantitative multi-analyte assay using molecular recognition at bioaffinity based binding sites.

Traditional immunodiagnostics have limited throughput and are restricted to screening for analyte species only known a priori. Protein chips could overcome this hurdle by presenting chemically and spatially multiplexed capture sites (micron-scale volumes containing protein docking sites immobilized in a support structure) for global sample analysis. Our work focuses on arraying commercially available antibodies, including antibodies with specificity for the *Bacillus* species and for the lethal and edema toxins of *Bacillus anthracis*. Fluorescently labeled analyte samples will be driven through the protein array using microfluidic fluid control, leveraging advantageous surface physics available at these short length scales. Work will be expanded to include analysis of native (unlabeled) analytes to eliminate confounding behaviors related to covalent analyte modifications required for labeling. Hyperspectral scanning and analysis techniques developed at Sandia will be employed, as will computational biology. While the concept of arrayed DNA and proteins is not new, no high-throughput, sensitive protein chip has been demonstrated as either a general experimental platform or a biodetector.

## Accomplishments

We developed capabilities for high throughput arraying of proteins and antibodies on derivatized substrates, using modified techniques initially developed for DNA microarray analyses. This will fulfill our first and second year milestones in the core technical areas (immobilization of active proteins, and imaging and quantitation of interactions). We developed and implemented robust substrate preparation, protein contact printing, substrate blocking, assay incubation, substrate washing, and imaging techniques and protocols. In addition to the two model systems evaluated in FY 2004, we presented and published results on a six element microarray for detection of toxins in buffer and serum systems.

The work conducted in FY 2005 resulted in the generation of isotherms and the extraction of affinity measurements for cholera toxin b-subunit, diphtheria toxin, anthrax lethal factor and protective antigen, *Staphylococcus aureus* enterotoxin B, and tetanus toxin c-fragment, all toxins or surrogates of interest to security considerations. We explored microcontact printing of antibodies on epoxide-functionalized glass substrates. We obtained detection limits in the ng/mL range in both buffer and serum systems, making the technique competitive with currently available assays for other proteins.

One limitation of a direct detection assay is that the assay requires that an antigen of interest be covalently modified. Such modifications to the analyte of interest are reported to suffer from irreproducibility issues requiring intensive normalization strategies during data work-up. To overcome this challenge, we implemented a ratiometric competition assay to detect unlabeled (native) toxin in solution. Information from the direct assays developed previously by our group was used to choose a suitable concentration of labeled toxin to act as a reporter for the presence of native toxin in solution. The native toxin competes with the labeled toxin for receptor binding. This competition, and hence the presence of the native toxin, is reported as a diminution in the fluorescent signal observed at an immobilized antibody's location.

Due to competition between the labeled toxin and the unlabeled analyte to be detected in the competition assay, the concentration of reporter toxin must be as low as possible without compromising signal quality. We chose reporter concentrations that were minimally greater than the limit of detection (LOD) determined in the direct assay. Experimentally, we observed the maximum fluorescence signal when the concentration of unlabeled protein (titrant) was zero: the fluorescence signal decreased sigmoidally towards the minimum signal as the concentration of unlabeled toxin increased from 350 pg/mL to 10 µg/mL.

We performed the competition assay with all six antigens, both in buffer and dilute bovine serum, with reporter concentrations nominally greater than the LOD determined in the direct assay. As with the direct assay, we normalized the data to generate values that were plotted against the total concentration of unlabeled protein in solution. The raw data were fit to the Langmuir equation. In this competition assay, the inflection point of the plot (where Theta_fit = 0.5) relates to the 50% inhibition constant, the point at which only 50% of the fluorescent reporter is bound by the surface immobilized antibodies in the presence of unlabeled inhibitor. The IC50 value is dependent on the concentration of fluorescent reporter, but with the previously determined dissociation constant (Kd) values, one can readily calculate the concentration-of-reporter-independent inhibition constant (Ki) using the equation Ki = IC50 ÷ (1 + [reporter]/Kd).

This is the first work reported in the literature regarding a planar microarray platform for detection of these important species. We measured protein concentration in sample volumes on the order of 50 µL, an important consideration for diagnostic systems.

## Refereed

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# Interaction of Proteins with Lipid Films

*67013*

*M. S. Kent, D. Y. Sasaki, L. J. Frink*

## Abstract

Understanding protein assembly at interfaces is of vital interest to a range of important technology areas at Sandia, including membrane signaling and recognition, toxin detection, mitigating the effects of toxins/biowarfare agents, and nanoscience. While techniques are well developed at Sandia to probe lipid mobility and aggregation within monolayers and bilayers, little capability currently exists to examine protein conformations at lipid membranes. Specific issues to be addressed are protein orientation and surface density; monolayer versus multilayer adsorption; conformational changes upon adsorption, including denaturization; the penetration of proteins into lipid membranes; the role of specific versus nonspecific interactions; and lateral order or crystallization. We apply neutron and x-ray scattering techniques to address these issues, along with more traditional experimental methods and calculations using fluid/polymer density functional theory. This combined approach is yielding new insights into protein-membrane interactions that could lead to the design of improved biosensing techniques, inhibitors and receptors for biological toxins of importance to national security, and smart biomimetic systems for nanoscience.

In FY 2005, we examined the binding of the tetanus toxin C-fragment to monolayers of dimyristoyl-*sn*-glycero-3-phosphotidylcholine mixed with 10 mol % of the ganglioside receptor GT1b, and then examined the effect of lowering the pH. We also performed initial studies of the binding of concanavelin A to a synthetic sugar-containing lipid and used a model synthetic system to study the effect of multiple interaction sites on the protein, on altering protein conformation and lipid phase behavior. In the final year, we will focus on the binding of the full tetanus toxin and the diphtheria toxin under a variety of conditions to both Langmuir monolayers and supported lipid bilayers. We will determine if conformational changes occur with a drop in pH, as proposed in the leading model explaining enzyme fragment penetration through cell membranes. We will also study the energetics of insertion of amphiphilic helical peptides into lipid membranes using density functional theory.

## Accomplishments

We determined that the tetanus toxin C-fragment binds to monolayers of dimyristoyl-*sn*-glycero-3-phosphotidylcholine mixed with 10 mol % of the ganglioside receptor GT1b with a binding constant in the range of 100–150 nanomolar. In that concentration range (and at pH 7.4), reflectivity data suggest that the bound protein retains its native state. No change in conformation was detected when the pH was dropped to 4.0. This is consistent with the hypothesis that it is the N-terminal region of the heavy chain that changes its conformation with pH to translocate the enzyme through the membrane.

With the histidine-metal ion binding system, we found that the presence of multiple specific binding sites on a protein lead to several effects that do not occur with single site binding. Multiple site binding leads to protein conformational changes and disruption of the lipid packing. By varying the metal ion within the headgroup, we covered a range of interaction energies spanning the transition from irreversible to reversible binding with histidines at room temperature.

We also found that placement of histidines along a helical peptide can have a significant effect on the adsorption behavior. The data to date suggest that a greater fraction of helicity is present in the bound state when the histidines are closer together.

We developed a method of tethering lipid bilayers onto solid supports using a specially synthesized tethering molecule and characterized the bilayers using fluorescence microscopy. We developed and completed fabrication of a liquid cell that can be used for both neutron reflectivity and fluorescence imaging. We also studied the pressure dependence of insertion of melittin into lipid monolayers in the gel state.

We initiated a neutron reflectivity study of diphtheria toxin bound to lipid monolayers containing negatively charged lipids. Diphtheria is similar to tetanus in that a drop in pH is believed to induce a conformational change that translocates the enzyme through the membrane.

In our effort toward a supported lipid bilayer platform for investigating protein-membrane interactions, we developed a tethered bilayer system with the associ-

ated microfluidics. The tethered bilayer is a lipid membrane that is immobilized to the substrate by a tethering lipid. The tether lipid was designed and synthesized with a hydrophobic cholesterol group at one end of an oligoethylene glycol and an amine group at the other. Amine coupling to a glycidoxy silane self-assembled monolayer provides the covalent linkage for the tether to the substrate. The oligoethylene glycol spacer is used to control the spacing of the bilayer off the substrate surface, while the cholesterol, which integrates into the membrane, acts as the anchor to hold the membrane in place. This year, we prepared glass slides and beads with the tethers, and evaluated the formation and stability of the bilayers on these surfaces in a fluidic cell using fluorescence microscopy. The bilayers formed a single bilayer over the surface with good stability observed over time. On the modeling side, we undertook a major redesign of molecular theory software in order to facilitate the calculations.

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M.S. Kent, H. Yim, D.Y. Sasaki, S. Satija, Y.-S. Seo, J. Majewski, E. Watkins, T. Gog, and I. Kuzmencko, "Protein Interactions with Lipid Membranes by Neutron and X-ray Reflectivity and GIXD," presented at the International Materials Research Congress, Cancun, Mexico, August 2005.

M.S. Kent, H. Yim, D.Y. Sasaki, J. Murton, S. Satija, J. Majewski, and I. Kuzmenko, "Protein Adsorption to Metal-Chelating Lipids: Conformational Changes and Insertion of Segments Due to Multiple Site Binding," presented at the Annual Meeting of the Protein Society, Boston, MA, August 2005.

A. Slade, A.E. Schroeder, C.M. Yip, and D.Y. Sasaki, "Investigating Tetanus Toxin-Membrane Interactions via In Situ Scanning Probe Microscopy and Fluorescence Leakage Studies," presented at the Biophysical Society Meeting, Long Beach, CA, February 2005.

D.Y. Sasaki, A.L. Slade, A.E. Schroeder, C.M. Yip, and J.S. Schoeniger, "Tetanus Toxin Activity on Lipid Membranes," presented at the New Mexico Institute for Mining and Technology, Socorro, NM, March 2005.

D.Y. Sasaki, A.L. Slade, A.E. Schroeder, C.M. Yip, and J.S. Schoeniger, "Analysis of the Binding and Pore Formation of Tetanus Toxin in Lipid Membranes," presented at the American Chemical Society Spring 2005 National Meeting, San Diego, CA, March 2005.

# New Technologies for Understanding Membrane Protein Recognition and Signaling

*67014*

*J. S. Schoeniger, P. Novak, R. B. Jacobsen, C. R. Behrens, A. M. Highley*

## Abstract

Structural elucidation of membrane proteins is difficult by standard techniques such as x-ray crystallography and nuclear magnetic resonance (NMR) due to the hydrophobicity of the proteins. Thus, development of alternative strategies is paramount. Due to the low concentration of sample necessary and the speed of the technique, chemical cross-linking and mass spectrometry can be used to extract distance constraints from membrane proteins and other hard-to-study proteins such as self-digesting proteases or aggregating proteins. These distance constraints can be used in combination with structural modeling to test structural hypotheses for membrane proteins. This project is directed toward improving the biochemical and analytical methods needed to identify cross-links, showing that diverse proteins can be investigated, and improving the fundamental interpretation and utility of cross-linking based structural studies.

In order for cross-links to be useful in constraining structural models, they must be identified. We demonstrated that advanced tandem mass spectrometry (MS/MS) techniques such as electron capture dissociation can be used to assign residues involved in cross-links between large hydrophobic peptides from bovine rhodopsin. We used these techniques to explore questions about structural mobility in rhodopsin. We also demonstrated that our cross-linking and MS methods can be applied to finding cross-links in recombinant human glycine receptors.

Recently, we quantified cross-linking reaction rates and demonstrated that these rates can be related to the Calpha separation of the reacting residues. Cross-linking rate measurements can now be used as actual distance measurements, not just as loosely-defined constraints or bounds. We are hopeful that this may become a general tool for measuring inter-residue distances in proteins complementary to established methods such as fluorescence resonance energy transfer, erythrocyte sedimentation rate, and NMR.

## Accomplishments

- *Methods for elucidating cross-links and protein structure:* Bovine rhodopsin is the standard experimental system for exploring the structure of G-protein coupled receptors (GPCRs). In previous years, we carried out extensive methods development on this system to allow identification of cross-linked peptides. However, the exact residues involved in the cross-links were difficult to resolve in some cases. Standard MS/MS techniques such as collision-induced dissociation (CID) were not able to identify certain fragments. However, we recently published findings that show quadrupole-Fourier transform mass spectrometry with electron capture dissociation can identify those fragments through sequencing, even in difficult species such as large cross-linked hydrophobic peptides. These results, combined with additional CID results obtained using a quadrupole-time-of-flight instrument, enabled us to elucidate a significant number of cross-links in bovine rhodopsin. The results allowed us to compare them with those predicted from the crystal structure and establish that portions of C-terminal regions of the protein have large mobility or differ from the crystal structure, in agreement with results from methods such as fluorine NMR and electron spin resonance.
- *Amino acid accessibility:* We previously demonstrated that amino acid reactivity with alkylating reagents could be profiled using the methods developed for cross-linked peptide identification. We developed a pulse-chase method that allows the pseudo-first-order rate constants of such alkylation reactions to be measured, allowing quantitative comparison of reactivity. Studies to quantify changes as a function of pH and structure are under way in rhodopsin.
- *Distance measurements:* Detailed consideration of cross-linking reaction mechanisms, as well as of the ionization properties of cross-linked proteins and peptides, allowed us to develop methods for the quantitative measurement of cross-linking reaction rates. Using kinetic theory and polymer theory, these rates can be related to the Calpha-Calpha distances of the reacting residues. We are using results for model helical peptides, the standard “spectroscopic ruler” used in

biophysics, to validate these methods, and we are extending the methods to measuring backbone distances in proteins.

## Refereed

P. Novak, W.E. Haskins, M.J. Ayson, R.B. Jacobsen, J.S. Schoeniger, M.D. Leavell, M.M. Young, and G.H. Kruppa, "Unambiguous Assignment of Intramolecular Chemical Cross-Links in Modified Mammalian Membrane Proteins by Fourier Transform-Tandem Mass Spectrometry," *Analytical Chemistry*, vol. 77, pp. 5101–5106, 2005.

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

*79745*

*R. Shediac, M. J. Ayson, S. Chhabra, J. S. Schoeniger, G. S. Chirica*

## Abstract

We propose to build 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 it is known that 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 or chemical 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 will consist of a micro-reactor, an amino acid separator, and an analyzer that 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 is expected to produce valuable intellectual property. This device has the potential to replace MS for bioagent detection and commercial applications (for proteomics and drug development), which together make up a market of > $100M/year. The device is a logical extension of protein based identification systems. Once demonstrated, it would serve as the foundation of a next generation of microfluidic detection platforms.

## Accomplishments

We made significant progress toward the development of a C-terminal enzymatic reactor. We determined that in-solution digestion of a peptide using a mixture of carboxypeptidases B and Y (CPBY) results in more efficient C-terminal cleavage than using either carboxypeptidase B (CPB) or carboxypeptidase Y (CPY) alone. CPY, CPB, and CPBY enzymatic reactors were generated by covalently immobilizing the enzymes onto porous silica beads using glutaraldehyde. We demonstrated that immobilized CPB, CPY, and CPBY reactors cleave 6 amino acids from the C-terminus of the peptide angiotensin in 5 minutes. Moreover, the CPBY reactor can cleave blocked C-terminal peptides. The amidated C-terminus of the peptide bombesin was cleaved with immobilized CPBY in 5 minutes, and a further 5 amino acids were released after 15 minutes. The CPBY reactor cleaves 3 C-terminal amino acids from the low molecular weight protein ubiquitin (approximately 8.6 kDa) within 5 minutes. We are currently optimizing reaction conditions for whole proteins. We developed a design for the microfluidic protein sequencing system that is based on the integration of discrete micro-reactor, amino acid separator, and detector modules using low dead-volume capillary-to-capillary connectors. In our design, protein will be delivered under pressure to the enzymatic or chemical reactor, and the resulting cleaved amino acid(s) will be separated from the truncated protein using size exclusion chromatography. The cleaved amino acid(s) will be labeled, passed into a capillary electrochromatography module, and detected via laser induced fluorescence using a µChemLab™ detection module.

# Tools for Characterizing Membrane Rafts and Toxin Interactions

*79746*

*T. M. Alam, G. P. Holland, M. J. Stevens*


## Abstract

This project is directed toward the development of nuclear magnetic resonance (NMR) spectroscopy tools that allow for the experimental measurement of the size and chemical composition of membrane rafts or microdomains. These tools will allow for the analysis of toxin-membrane interactions and the resulting biochemical activity. This research involves the development of NMR experimental spin-diffusion and dipolar re-coupled pulse sequences that can detect and measure membrane raft domain size and dynamics and extend these techniques to probe toxin binding at the membrane surface. The first year of this project resulted in the development of several new NMR sequences directed toward understanding molecular interactions within model biomembranes.

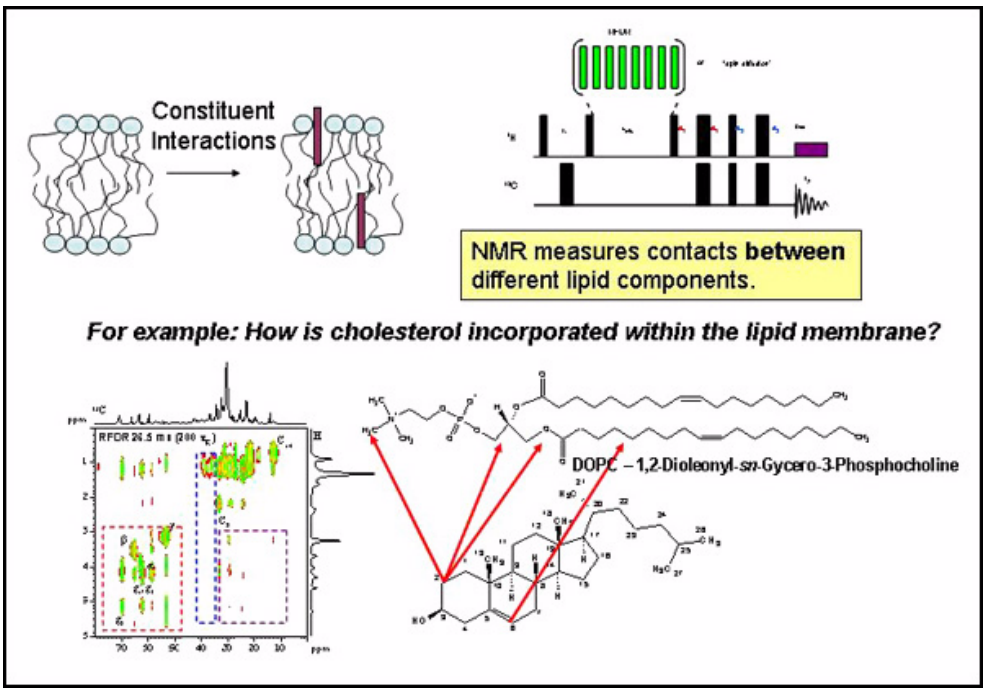

## Accomplishments

FY 2005 research involved the development and implementation of new and novel NMR pulse sequences that allow us to probe specific molecule-molecule interactions, structural dynamics, and disorder. They also enable the measurement of domain size in raft forming biomembrane systems. We made excellent progress on the FY 2005 milestone and subtasks detailed below.

(1) We obtained high resolution $^{1}$H, $^{13}$C, and $^{31}$P magic angle spinning (MAS) NMR spectra for these model membrane systems as a function of temperature. Results allowed us to make assignment of the various resonances within the molecules, enabling more complex experiments as described below.

(2) In combination with MAS NMR studies, we also obtained excellent wide line static $^{31}$P NMR spectra that allow the different dynamics of moieties to be experimentally measured. Even these simple experiments produced some interesting results.

(3) For the di-oleonyl-phosphocholine/sphingomyelin/cholesterol (DOPC/SM/CHOL) model membrane system, we demonstrated that the different lipid species could be resolved by $^{31}$P MAS NMR. This work is the first demonstration of the use of $^{31}$P MAS NMR to resolve the chemical shielding anisotropy (CSA) tensor for different lipid species as a function of temperature and composition. This is a powerful capability, and we are preparing the results of this work for publication.




*NMR tools for biomembrane molecular contacts.*

(4) We obtained the first two-dimensional (2-D) $^{1}H$-$^{31}P$ MAS NMR exchange correlation spectra for a multi-component biomembrane. We measured differential $^{1}H$-$^{1}H$ spin exchange rates and water contacts and are exploring them further.

(5) We demonstrated the use of 2D $^{1}H$-$^{13}C$ INEPT (insensitive nuclear enhancement by polarization transfer) based spin diffusion experiments. This provided some of the first NMR measurements of lipid/cholesterol contacts at the molecular level.

## Other Communications

T.M. Alam and G.P. Holland, "Probing Biomembrane Organization Using INEPT Based MAS NMR Exchange Experiments," presented at Rocky Mountain Analytical NMR Conference, Denver, CO, August 2005.

G.P. Holland and T.M. Alam, "Probing Organization in Model Lipid Biomembranes with INEPT Based Spin Diffusion and Selective Excitation 31P MAS NMR," presented at the American Chemical Society National Meeting, San Diego, CA, March 2005.

T.M. Alam and G.P. Holland, "The Development of 1H-13C INEPT MAS NMR Spin Diffusion Experiments for Studying Biomembranes," presented at the Biophysical Society Annual Meeting, Long Beach, CA, February 2005.

# Integrated Nanosystems for Monitoring Cell Signaling Proteins

*79747*

*B. C. Bunker, M. Okandan, J. C. Harper, M. Farrow, W. G. Yelton, G. D. Bachand, J. A. Voigt*

## Abstract

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 will be 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. This will form the basis for enduring partnerships in which Sandia's expertise in microsystems will be coupled to strong molecular biology groups at other institutions, including Los Alamos National Laboratory, the University of Texas Southwestern Medical Center (UTSW), and the National Biocontainment Laboratories located at the University of Texas Medical Branch (UTMB), Galveston. Such partnerships will help 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: (1) Protein Capture Monolayers – We are developing surfaces that can be electrochemically programmed with antibodies, allowing for highly selective adsorption of specific proteins on top of an underlying sensor array. (2) Sensor Elements – We are developing porous films that can function as ultracapacitors or electrochemical sensors. The currents generated in such films vs. applied voltage are extremely sensitive to the presence of adsorbed proteins via a pore blocking mechanism. (3) 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. (4) Biological Testing – We are testing the fluidic system with white blood cells and are detecting the production of cytokines such as interleukins.

## Other Communications

## Accomplishments

- *Programmable Surfaces for Protein Capture:* We synthesized self-assembled monolayers based on a cyclodextrin host with multiple thiol groups to tether them to gold surfaces. We functionalized ferrocene guest molecules with biotin to facilitate attachment of antibodies. Electrochemical and quartz crystal microbalance experiments show that reduction and oxidation of the functionalized ferrocene result in reversible adsorption and desorption, indicating that surface programming is feasible.
- *Ultracapacitor Sensors:* We prepared and tested silica films containing monodisperse 8 nm pores as highly sensitive ultracapacitor and electrochemical sensor elements. Films functionalized with biotin detected as little as 10 femtomoles of streptavidin. The results suggest that sensors based on ultracapacitors may be capable of detecting less than 10 protein molecules on a 1 µm x 1 µm electrode.
- *Microfluidic Platforms:* We designed a microfluidics platform to capture cells, support cellular functions, and incorporate the programmable surfaces and ultracapacitor sensors described above. The platform contains a central patch clamp for cell capture, fluidics ports at the corners for feeding fluids to and extracting fluids from the cell capture zone, and electrode arrays that can apply voltages to ultracapacitor sensors.
- *Functionalized Particles:* We functionalized fluorescent quantum dots with polylysine to facilitate the penetration of these optical labels into living cells via endocytosis. Fluorescence microscopy experiments show that these quantum dots penetrate the cell wall in less than five minutes. However, the quantum dots also penetrate vesicles that are internal to the cell, removing them from intercellular fluids.

## Refereed

Q. Huo and J. Liu, "Self-Assembled Nanoporous Electrodes for Sensitive and Labeling-Free Biomolecular Recognition," to be published in *Applied Physics Letters.*

B.C. Bunker, "Electrochemically-Switchable Surfaces for Programmable Bio-Sensors," presented at the

American Physical Society March Meeting, Baltimore, MD, March 2006.

# Advanced Microscopy: Time Resolved Multi-Spectral Imaging of Single Biomolecules

*79748*

*D. W. Chandler, C. C. Hayden, A. K. Luong, C. C. Gradinaru*

## Abstract

Sandia recently developed the ability to acquire images through a confocal microscope that contain, for each pixel, the simultaneous fluorescence lifetime and spectra of multiple fluorophores within that pixel. We demonstrated that our system has the sensitivity to make these measurements on single molecules. This project takes advantage of this technology and builds on it to create the capability for multiplexed single molecule microscopy of biologically important systems. The spectra and lifetimes of fluorophores bound to complex molecules contain a wealth of information on the conformational dynamics and local chemical environments of the molecules. However, the detailed record of spectral and temporal information our system provides from fluorophores in single molecules has not been previously available. Therefore, this research involves studies of relatively simple molecular systems to obtain a basis for understanding how fluorophores report on the chemistry of more complex biological molecules.

## Accomplishments

We studied several interesting biomolecular systems. We chose relatively simple molecular systems so that we could use the unique capabilities of our detection system to study the interaction between molecular environments and important fluorophores such as fluorescent proteins and commonly used fluorescence resonance energy transfer (FRET) fluorophores. FRET is used in many biological applications as a measure of the distance between fluorescently labeled molecules.

In collaboration with Professor Haw Yang at the University of California, Berkeley, we have studied single pair FRET (spFRET) using short polyproline peptides with 12 and 24 units as molecular spacers between donor and acceptor fluorophores. In the literature, polyproline, assumed to be rigid, was used as a spacer to check the dependence of the FRET efficiency on the donor-acceptor distance. In our experiments, the polyproline molecules are immobilized on a PEG-biotin surface. The sample is excited at 532 nm with 7 ps pulses from a mode-locked laser. Correlated fluorescence emission spectra and decays for spFRET are detected. The fluorescence spectrum has two clearly separated peaks, at around 565 nm from the excited donor fluorophore and at 670 nm from the acceptor, indicating efficient FRET. In our data, the emission time of every photon is recorded simultaneously with the emission wavelength, allowing us to construct, post acquisition, any combination of time gated spectra or wavelength gated decays. We are also able to apply powerful techniques of correlated photon statistics to maximize the information obtained on the system.

The coupled spectra and time decays reveal the dynamics of the fundamental electronic energy transfer process. Time histories of the spectra and decays record fluctuations of the FRET efficiency due to conformational changes and fluctuations in the spectral characteristics of the fluorophores. The spFRET distance measurements show that the effective length of the polypeptides is less than expected, demonstrating that they are not fully rigid. These spFRET measurements also show different efficiencies for different molecules, suggesting the possibility of multiple conformations. However, because we measure full spectral and temporal information about the fluorophores, we could determine that the differences in FRET efficiencies result from differences in donor fluorescence lifetimes. This is presumably due to interactions between the fluorophores and the surface on which they are immobilized. Results from our experiments were published in a feature article in the *Journal of Physical Chemistry.*

Fluorescent proteins are often used in studies of cellular processes because they can be introduced into cell proteins through genetic mutation. We studied two variants, green fluorescent protein (GFP) and yellow fluorescent protein (YFP). These proteins have multiple ground state configurations with different fluorescence characteristics. Understanding the variations in fluorescence characteristics is important because they can provide information on the chemical environment around the fluorescent protein, but they can also interfere with FRET measurements that use fluorescent proteins. We have measured the time variations in the fluorescence spectra and lifetimes of many individual

GFP and YFP molecules. These data will enable better understanding of the mechanisms and time scales for transitions between the ground state configurations of GFP and YFP, and determine the fluorescence properties of specific configurations.

Quantum dots are becoming increasingly important as fluorophores for biological studies because of their tremendous resistance to photobleaching. However, quantum dots exhibit some non-ideal behaviors; the best known is blinking, in which the fluorescence turns on and off. In collaboration with Professor Yang, we studied the intensity fluctuations of biologically functionalized quantum dots. We measured the correlation between changes in the fluorescence intensity, lifetime, and spectrum of individual quantum dots. These are measurements for which our apparatus is uniquely suited. The fluorescence properties are found to be strongly correlated. These studies will help determine which characteristics of the quantum dots are responsible for their non-ideal optical behavior.

## Refereed

A.K. Luong, C.C. Gradinaru, D.W. Chandler, and C.C. Hayden, "Simultaneous Time- and Wavelength-Resolved Fluorescence Microscopy of Single Molecules," *Journal of Physical Chemistry B*, vol. 109, pp. 15691–15698, 2005.

## Other Communications

C.C. Hayden, A.K. Luong, C.C. Gradinaru, and D.W. Chandler, "Time-Resolved Multispectral Imaging of Single Molecules," presented at Frontiers in Optics 2004/Laser Science XX, Rochester, NY, October 2004.

C. Gradinaru, A.K. Luong, C. Hayden, and D. Chandler, "Spectrally Dispersed FLIM of Single Molecules: Probing Local Environment and Measuring FRET," presented at Frontiers in Optics/Laser Science XX, Rochester, NY, October 2004.

# DNA Based Intelligent Microsensors for Genetically Modified Organisms (GMOs)

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

## Abstract

Advances in genetic engineering make weaponized forms of bioagents an imminent threat to our national security. Existing biosensor systems are promising, but currently lack the ability to intelligently sense pathogens of interest in complicated backgrounds or to detect genetically modified organisms (GMOs). It is imperative to move beyond static sensor systems and develop robust, "smart" detection systems that leverage molecular computation to systematically identify biological pathogens and their genetically modified variants in the presence of non-lethal agents.

Deoxyribozyme gates, a newly developed technology, can be combined in numerous configurations (AND, OR, AND-NOT gates) to achieve a desired response in the presence of multiple biological markers. We propose to leverage their computational ability, demonstrated by Stojanovic and Stefanovic in a dynamic tic-tac-toe game [1], to develop differentiating capabilities for designing DNA-based biosensor systems that detect mutations in target oligonucleotides, a necessity for GMO detection. Project milestones include: (1) Develop algorithms for hierarchical Boolean networks for pathogen detection in complex environments, gate-level cross talk minimization, statistical signal analysis; (2) Combinatorial design and development of efficient catalyst/oligomer libraries; (3) Design catalytic molecular beacons and target oligonucleotides and demonstrate fluorescence reporting from immobilized sensing elements; and (4) Develop microfluidics and micro-optics technology for sensing results of molecular Boolean networks.

## Accomplishments

The primary goal for this year was to demonstrate the feasibility of the deoxyribozyme sensor to sense deoxyribonucleic acid (DNA) targets with the same fidelity as current DNA sensing technology. Our accomplishments are summarized below.

(1) We developed a Xyce-based simulation of the basic gate structures described by Stojanovic and Stefanovic [1]. The DNA gates are combined with OR gates to simulate a computational well. Nine computational wells are used to simulate Stojanovic and Stefanovic's tic-tac-toe molecular automaton. Simulation results are behaviorally consistent with the fluorescence output order as reported.

(2) Using experimental data produced by introducing single and double mutations in the probe sequence, we are exploring correlations between free energy of the target-probe complex and the measured fluorescence. Experimentally generating mismatch data is prohibitive beyond two mutations. Therefore, we investigated molecular dynamics based approaches to perform "in silico" experiments that accurately replicate the in vitro behavior of the gates.

(3) We designed and synthesized two deoxyribozyme gates, YESiA (E6) and iAANDiB (8-17).

(4) We successfully demonstrated detection of target DNA at concentrations matching or exceeding the detection limits of current DNA sensing technology. Using a 55 µl detection volume containing the YESiA gate (100 nM) and the tetramethylrhodamine substrate (2.5 µM), our detection limit was between 1.25 femtomoles and 500.5 attomoles of target DNA (~3–7.5 x $10^8$ molecules).

(5) Using a 55µl detection volume containing the YESiA gate (100 nM), TAMRA substrate (1 µM), and input sequences (2 µM), we were able to distinguish the target from mismatched sequences within 3 minutes. This trend continued for more than 3 hours.

[1] M. Stojanovic and D. Stefanovic, "A Deoxyribozyme-Based Molecular Automaton," *Nature Biotechnology*, vol. 21, pp. 1069–1074, August 2003.

# Chemical and Earth Sciences

## Sequestration of Pathogens on Nanoengineered Surfaces

*52538*

*M. S. Kent, G. S. Chirica, D. L. Huber, J. G. Curro, M. D. Nyman*

### Abstract

Recent events are heightening awareness of the vulnerability of US water resources to bioterrorism. There is a critical need for both rapid sensing and improved mitigation technologies. Our goal was to develop materials that sequester pathogens based on chemical interactions, rather than size alone. These materials are targeted for both sensing and improved water treatment applications. For sensing, our goal was a preconcentrator for a biosensor system that will capture and concentrate pathogens from water streams, and then release them in a concentrated pulse. We developed a method based on adsorption to a high surface area media through nonspecific interactions, followed by release by elution with an appropriate release agent. For mitigation, one goal was to develop filtration materials for bacteria and viruses based on poly(N-isopropylacrylamide) (PNIPAM) that allow for lower pressures, higher flow rates, and improved efficiency relative to current ultrafiltration technology. PNIPAM is the best known temperature responsive polymer, possessing a lower critical solution temperature (LCST) at 31 °C. Under optimal conditions, grafted PNIPAM chains and PNIPAM gels undergo a dramatic collapse transition near the LCST, which can be used to open and close pores to achieve temperature dependent filtration. Another goal was to develop polyoxometalate (POM) materials that inactivate viruses. POMs are anionic, metal-oxo clusters with 6 to 30 metal octahedra of W, Mo, Nb, V, or Ta (d0), often assembled around 1 or 2 central tetrahedra. POM clusters have a charge ranging from approximately -4 to -20, and are known to interact strongly with viruses and some bacteria through electrostatic interactions. We also investigated various POM materials with respect to their interactions with bacteria.

### Accomplishments

We gathered data to determine the maximum concentration factor for a fixed range of inlet bacterial concentration for *Cobetia marina* (CM, Gram-negative, vegetative cells in log phase) and for *Bacillus atrophaeus* (BA, Gram-positive, vegetative cells in stationary phase and spores). The latter is in the same genus as *Bacillus anthracis*, indicating that the method likely applies to concentrating samples of anthrax spores from aerosol collectors. For vegetative cells, we achieved concentration factors in excess of 1000X. For spores, we achieved concentration factors approaching 300X.

We completed experimental and theoretical studies of the conformational transition of PNIPAM grafted chains. We discovered that the magnitude of the conformational change with temperature varies strongly with surface density and molecular weight. We observed the maximum effect for intermediate grafting density and high molecular weight.

We used self-consistent field theory to calculate the force between tethered PNIPAM chains and a neutral surface at 20 °C and 40 °C. The range of the force was much larger at 20 °C (below the LCST transition) than at 40 °C. The theoretical results were in good qualitative agreement with atomic force microscopy measurements in the literature.

We employed a swelling theory published in the literature and generalized it to polymer gels with LCST behavior. Using data published in the literature on the phase diagram of aqueous PNIPAM solutions, we predicted the swelling ratio of PNIPAM gels as a function

of temperature, crosslink density, and solvent quality. Very large changes in volume (factors of 20 or greater) are predicted when the gels are heated or cooled through the LCST.

We synthesized PNIPAM grafted layers optimized for maximum conformational change onto glass beads and also onto diatomaceous earth (DE) to achieve temperature-dependent filtration materials to capture bacterial and viral particles. We achieved temperature-dependent filtration of bacteria with PNIPAM synthesized onto 300-400 µm glass beads. However, this only worked for very thick layers that included a substantial amount of adsorbed polymer in addition to grafted polymer, and the layers were not stable in water over the long term. Layers rinsed free of adsorbed chains were too thin to capture bacterial cells, so we shifted emphasis to capturing viral particles. We tested PNIPAM synthesized onto DE for the capture of 500 Å fluorescent polystyrene (PS) spheres as substitutes for viral particles. The PNIPAM grafted layers appeared to be sufficiently thick to block the passage of PS spheres through the pores even in the collapsed state. We made a second attempt in which we synthesized the PNIPAM at a lower grafting density.

We developed a protocol for varying the thickness of crosslinked PNIPAM gels for temperature dependent filtration of bacteria or viruses.

We formulated a wide variety of POMs and deposited them as coatings. These materials varied in charge, chemical stability, composition, and geometry.

We tested the affinity of influenza A, swine influenza virus, bovine coronavirus, and bovine rotavirus to various functionalized surfaces (PNIPAM of various surface densities and molecular weights, polystyrene, octadecyltrichlorosilane, acid-cleaned bare glass, polyethyleneglycol, a quaternary ammonium compound, and various POMs) at Kansas State University. Surprisingly, the viruses showed comparable affinity to this very wide range of surface treatments.

We investigated the temperature-dependent binding and release of tobacco mosaic virus onto PNIPAM coated surfaces.

We determined the cytotoxicity of various synthesized POMs to Madin-Darby canine kidney cells. The POMs were relatively noncytotoxic at concentrations as high as 0.1 mM.

We determined the antiviral efficacy of various POMs with respect to influenza A, bovine enterovirus, and bovine rotavirus. We found significant antiviral efficacy with respect to influenza A at dilute concentrations. We found less efficacy for bovine rotavirus and bovine enterovirus. This may be due to the fact that the latter are nonenveloped, whereas influenza A is an enveloped virus.

## Refereed

H. Yim, M.S. Kent, S. Mendez, G.P. Lopez, S. Satija, and Y. Seo, “Effects of Grafting Density and Molecular Weight on the Temperature-Dependent Conformational Change of Poly(N-isopropylacrylamide) Grafted Chains in Water,” submitted to *Macromolecules*.

H. Yim, M.S. Kent, S. Satija, S. Mendez, S.S. Balamurugan, S. Balamurugan, and G.P. Lopez, “Evidence for Vertical Phase Separation in Densely Grafted, High Molecular Weight Poly(N-isopropylacrylamide) Brushes in Water,” submitted to *Physical Review E*.

S. Mendez, J.G. Curro, J.D. McCoy, and G.P. Lopez, “Computational Modeling of the Temperature-Induced Structural Changes in Poly(n-isopropyl acrylamide) with Self-Consistent Field Theory,” *Macromolecules*, vol. 38, p. 174, 2005.

T.S. Phely-Bobin, J.S. Weems, D.L. Huber, and M.S. Kent, “Separation of Bacterial Species Using Poly(N-isopropylacrylamide)- Modified Silica Based Hydrophobic-Interaction Chromatography (HIC),” submitted to *Analytical Chemistry.*

## Other Communications

M.S. Kent, J.S. Weems, H. Yim, J. Curro, D. Huber, T. Phely-Bobin, J. Fruetal, G. Chirica, G.P. Lopez, L.K. Ista, J. Bieker, S. Kapil, and D. Oberst, “Sequestration of Bacteria and Viruses for Preconcentration and Water Purification,” presented at Kansas State University School of Veterinary Medicine and Veterinary Diagnostic Center, 2005.

M.S. Kent, H. Yim, S. Mendez, G.P. Lopez, and S. Satija, “Temperature-Dependent Conformational Changes of PNIPAM Grafted Chains in Water: Effects of Molecular Weight and Grafting Density,” presented at the American Physical Society, Los Angeles, CA, March 2005.

J.G. Curro, “Computational Modeling of the Temperature-Induced Structural Changes of Tethered Poly(N-isopropylacrylamide),” presented at the American

Physical Society, Los Angeles, CA, March 2005.

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D.L. Huber, B.C. Bunker, R.P. Manginell, T.S. Phely-Bobin, and G.D. Bachand, “Reversible Adsorption on Poly(N-isopropyl acrylamide) and Implications to Biotechnology,” presented at the International Conference on the Frontiers of Polymers and Materials, Cancun, Mexico, April 2005.

# A Combined Preconcentrator and Sensor for Live Water Borne Pathogens

*52539*

*E. B. Cummings, B. A. Simmons*

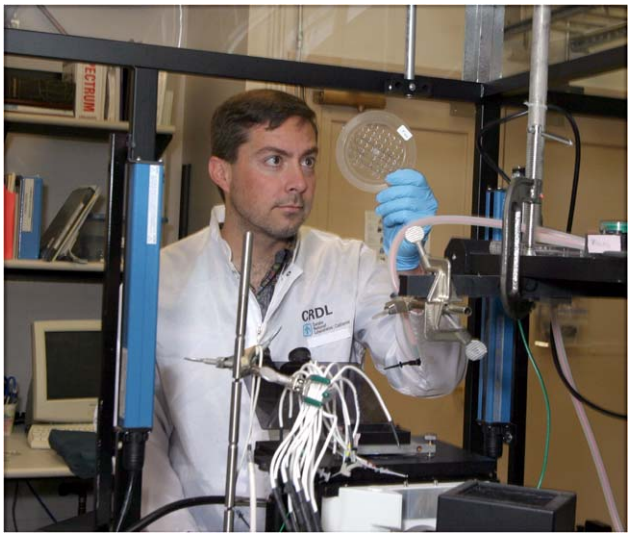
## Abstract

The insulator based electrodeless dielectrophoretic (iDEP) concentrator concept employs a sequence of physically specific interactions: (1) During collection, a moderate applied electric field dielectrophoretically traps particles in size specific zones of the device containing a high volume. nonuniform array of insulating posts, while other solutes and water flow unimpeded to a waste stream. (2) Trapped particles within a selected size range are released in a concentrate to a medium volume array having a direct current (DC) offset: Particles that are specific to the internal physique of the particles are retrapped in zones of this secondary device. (3) Selected particles are then released to a tertiary low volume dielectrophoretic sieve bearing an electric field generated conductivity gradient to further filter and select particles that physically respond to osmolality and conductivity, a live-particle signature. (4) A sharply pulsed electric field lyses the selected cells, releasing their contents to a canonical microfluidic separation channel. This will be the first device that uses physics that occur at the microfluidics scale to take large sample volumes to small sample volumes.

Our data indicate that bacteria trap at lower field strengths than spores, which trap at lower electric field strengths than viruses. We also demonstrated bacterial and particle trapping in plastic devices. We characterized the existing, lowest volume concentrator stage for removal efficiency and concentration factor.

## Accomplishments

Having proven the effectiveness of the iDEP methodology for selectively concentrating organisms in low-glass microsystems, we set our focus on the engineering of devices that robustly and economically handle much higher volumetric flow rates, according to the needs of water analysis. To this end, we (1) studied how devices behave with combined electrokinetic and pressure driven flow; (2) tested how devices behave with low frequency (< 10 kHz) alternating current (AC) excitation and pressure driving; (3) tested how devices behave with static and dynamic coatings to eliminate or minimize particle sticking; (4) created a process to make polymeric iDEP devices and studied how these devices perform compared to glass devices; (5) studied and filed a patent for a new class of iDEP devices that utilize a transverse membrane, mesh, or weave to produce spatially non-uniform fields to drive DEP; and (6) studied time domain field sequencing techniques for enhancing the performance of simple post array designs. As a result of these studies, we successfully scaled throughput with a single conventional device into the tens of milliliters per hour range. We found a static coating that adheres well to our polymer surface and does not dramatically affect electrokinetic particle flow, but is very effective at eliminating surface retention of both polystyrene and biological particles. Furthermore, we demonstrated our novel transverse membrane geometry to be effective at flow rates up to nearly 300 ml/hour with excellent scalability to much higher flow rates.



*Researcher Eric Cummings examines an insulative dielectrophoresis prototype that is used to separate live from dead water borne pathogens.*

We have made a portable, computer controlled or self-sequencing battery powered iDEP system with a fixture that can easily be used as a tool for customers to evaluate the effectiveness of iDEP for their applications. This project generated a Cooperative Research and Development Agreement (CRADA) with a major homeland security contractor, and generated a consid-

erable amount of interest among commercial customers and federal agencies, including the Awwa Research Foundation, which funded an iDEP water monitoring hardware development and testing project.

## Refereed

G.J. McGraw, R.V. Davalos, J.D. Brazzle, J.T. Hachman, M.C. Hunter, J.M. Chames, G.J. Fiechtner, E.B. Cummings, Y. Fintschenko, and B.A. Simmons, "Polymeric Microfluidic Devices for the Monitoring and Separation of Water-Borne Pathogens Utilizing Insulative Dielectrophoresis," presented and in *Proceedings of the SPIE, Micromachining and Microfabrication Process Technology X*; Mary-Ann Maher, Harold D. Stewart, Eds., January 2005, vol. 5715, pp. 59–68.

## Other Communications

E.B. Cummings, B.A. Simmons, R.V. Davalos, G.J. McGraw, B.H. Lapizco-Encinas and Y. Fintschenko, "Fast and Selective Concentration of Pathogens by Insulator-Based Dielectrophoresis," presented at the 230th ACS National Meeting, Washington, DC, Fall 2005.

Y. Fintschenko, et al., "Microfluidics Approaches to Pathogen Detection," presented at LabAutomation 2005, San Jose, CA, Winter 2005.

# Geophysical Subsurface Imaging and Interface Identification

*53586*

*C. J. Weiss, P. B. Bochev, D. M. Day, R. S. Tuminaro*

## Abstract

We addressed the problem of imaging complex three-dimensional structures in the subsurface through geophysical exploration technologies. In particular, we are interested in improved imaging methods for targets whose perimeter is better described by a sharp interface or discontinuity in material properties (for example: underground facilities, lithologic horizons, and groundwater tables).

Geophysical imaging relies on the mathematical process called inversion. To stabilize the process, a smooth model constraint is typically imposed on the subsurface geophysical properties (conductivity, velocity, density, etc.) such that they are only allowed to vary smoothly. While such constraints can eliminate unrealistic geological models, they come with a price of reduced model resolution, seriously limiting the applicability and relevance of geophysical imaging technologies for national security concerns. Electromagnetic applications will be emphasized; however, the scope of the methodology is much broader and pertinent to other geophysical applications, medical imaging, and the nondestructive evaluation of materials in engineering.

We are implementing a new type of geophysical inversion for interface identification. It will enable the location of underground structures and facilities, oil and gas deposits, and environmental site characterization at a scale not previously possible. Construction of the interfaces will require new optimization procedures and massively parallel linear and nonlinear solver tools developed under the Accelerated Strategic Computing program. Exact sequences of finite element methods will be used to discretize Maxwell's equations. Optimal algebraic multigrid solvers will be extended to handle variable conductivity and complex coefficient matrices. To obtain reliable and sharp interfaces, smooth reconstructions of the interface will first be used for the initial topology estimate, followed by well-posed parameterization of the surfaces based on interface tracking, fictitious domains, and level set approaches.

## Accomplishments

We developed and tested a moving interface, sharp boundary inversion algorithm for recovery of the three-dimensional distribution of control variables in materials stimulated by remote sensing phenomena and governed by elliptic partial differential equations. In particular, we examined the recovery of a "disk-like" feature through a numerical analogue to the direct current resistivity sounding and controlled source electromagnetic sounding methods. Initial results reveal that simple translations of the "disk" feature are recoverable through the inversion algorithm. However, there was insufficient time to test the robustness of the algorithm to more complicated or "diabolical" starting models. Other accomplishments include a C++ interface for the electromagnetic sounding forward algorithm to facilitate integration with Sandia optimization libraries. Also, in response to interest by the oil and gas industry, we have requested copyright assertion for the electromagnetic forward modeling code.

*Specific milestones and achievements*

- Implement adjoint based inversion using Moocho: We examined and demonstrated recovery of test structures from synthetic data.
- Implement sharp interface optimization tools: We implemented surface representation with the GTSURFACE discretization library.
- Demonstrate scalability of multigrid linear solvers applied to unstructured finite element discretizations of realistic geologic structures: We temporarily shelved finite element solvers in favor of finite difference solvers that are more tailored to geophysical exploration scenarios, and tested in such scenarios. We demonstrated linear scaling of the linear solver (QMR) for finite difference algorithms on multiple platforms for both matrix free and stored matrix formalisms.
- Apply forward modeling and inversion algorithms to field data: We attempted to apply forward modeling inversion algorithms to field data; however, limitations in computational resources limited application to synthetic data only.

## Refereed

D. Day and M. Heroux, "Roots of Polynomials Expressed in Terms of Orthogonal Polynomials," to be published in *SIAM Journal of Numerical Analysis*.

K. Key and C. Weiss, “Adaptive Finite Element Modeling Using Unstructured Grids: The 2D Magnetotelluric Example,” submitted to *Geophysics.*

K. Key, S. Constable, and C. Weiss, “Magnetotelluric Analysis of the Gemini Prospect, Gulf of Mexico,” to be published in *Geophysics.*

S. Constable and C. Weiss, “Controlled-Source Marine Electromagnetics: The Limits of 1D Analysis,” to be published in *Geophysics*.

# Poroelastic Wave Propagation Modeling and Inversion

*69198*

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

## Abstract

Sedimentary and igneous rocks, sands, soils, and certain man-made materials (concrete) consist of a solid framework surrounding a microscopic pore or fracture system saturated with fluids. The fluid phase may exist in a gaseous (air, $CO_2$, methane) and/or liquid (water, brine, oil) state. Movement of a viscous fluid within a spatially connected pore or fracture geometry is an important physical mechanism for attenuation and dispersion of seismic waves propagating in typical earth materials.

Three-dimensional (3-D) seismic wave propagation within a heterogeneous, isotropic poroelastic medium is numerically simulated with an explicit, time-domain, finite-difference (FD) algorithm. A system of 13 coupled first order partial differential equations (PDEs) is solved for the velocity vector components, stress tensor components, and pressure associated with solid and fluid constituents of the composite medium. Numerical techniques yielding high accuracy in 3-D elastodynamic solutions (staggered temporal and spatial storage, high order differencing) are utilized. The computational cost (memory and execution time) of poroelastic modeling is substantially greater than equivalent sized elastic or acoustic simulations. A massively parallel algorithm implementation, utilizing the spatial domain decomposition strategy, allows efficient investigation of large scale earth models and broadband wave propagation.

Inversion of recorded seismic data to infer the 3-D distribution of poroelastic medium parameters is a nonlinear inverse problem. The solution procedure entails iterative refinement of an initial model estimate until observed and predicted (computed) data agree to within an acceptable tolerance. A novel optimization methodology called simultaneous analysis and design (SAND) efficiently solves both forward and inverse equation systems simultaneously.

Accurate and realistic poroelastic wave propagation modeling and inversion capabilities will benefit the petroleum industry (for oil and natural gas exploration, production, and reservoir characterization purposes) and have numerous additional applications involving remote detection and monitoring of subsurface fluids and flow ($CO_2$ sequestration, shallow contaminant plume migration, hydrological exploration, earthquake source modeling).

## Accomplishments

The primary goal of this project is to develop robust computational algorithms for solving both the forward and inverse poroelastic wave propagation problems in three spatial dimensions (3-D). We derived a system of 13 coupled, nonhomogeneous first order PDEs describing linearized (infinitesimal deformation) wave propagation through a porous, fluid-saturated solid from fundamental principles of Biot theory. Utilizing a volume averaging procedure, we obtained particularly advantageous PDEs governing the phase-averaged solid and fluid particle velocity vector components, solid stress tensor components, and fluid pressure. We subsequently implemented explicit, time-domain numerical solution algorithms in both serial and massively parallel computational environments. The solution methodology adapted state-of-the-art FD techniques previously utilized in 3-D elastodynamics to the poroelastic situation. Thus, the 13 dependent variables are stored on staggered spatial and temporal grids. We employed fourth order, centered, staggered, spatial FD operators to obtain high accuracy, maintain numerical stability, and minimize numerical dispersion artifacts.

We mathematically developed and numerically implemented several improvements to the basic time-domain FD approach within the serial version of the algorithm. These improvements are designed to enhance seismological realism, provide additional user flexibility, and simultaneously increase accuracy and reduce the computational burden (both memory and execution time) associated with 3-D poroelastic forward modeling. Although the serial algorithm provides a convenient platform for developing and evaluating these code enhancements, we will ultimately introduce all improvements into the parallel version of the forward modeling algorithm. The parallel algorithm utilizes a spatial domain decomposition strategy. Portions of the 3-D model and wavefield are allocated to separate processors with dedicated memory. FD calculations take place concurrently within all processors, and

message passing is used to communicate necessary information for updating the wavefield variables between adjacent subdomains/processors. This approach optimizes parallel scalability.

Ongoing FD algorithm improvements designed to enhance seismological realism and flexibility entail implementing (1) realistic seismic energy sources characterized by force density vectors and moment density tensors; (2) seismic receivers sensitive to solid, fluid, and composite (porosity-weighted superposition of solid and fluid) medium motions, as well as pressure; (3) timeslice displays for wavefront visualization; (4) superior 3-D poroelastic model construction software; (5) absorbing boundary conditions for suppressing grid flank reflections; and (6) a vanishing stress/pressure boundary condition on a plane horizontal surface for mimicking real world seismic conditions at the air/earth interface.

Inversion of recorded seismic data to infer the 3-D spatial distribution of poroelastic parameters is a nonlinear inverse problem. The solution procedure entails iterative refinement of an initial estimate of an earth model until predicted (i.e., calculated) and observed data agree to within an acceptable tolerance. We utilize a weighted least-squares measure of misfit. With each iteration, we obtain updates to the current model parameters by constructing and solving a large system of linear algebraic equations. A SAND numerical inversion approach efficiently solves the forward and inverse problems simultaneously with the linear system.

We implemented several significant generalizations to the current 2-D inversion procedure in order to make the process more applicable to realistic seismic data acquisition scenarios. These involve (1) increasing the spatial dimensionality to 3-D, (2) recovery of four poroelastic moduli, (3) utilizing realistic data acquisition geometries consisting of a limited and sparse distribution of sources and receivers, (4) introducing numerical regularization into the process in order to resolve model parameter ambiguity, and (5) introducing synthetic noise in order to quantify algorithm stability and accuracy. We are currently upgrading the present Matlab-based code to an object oriented parallel code, written in C++ and sited within the Trilinos numerical analysis framework. In order to objectively evaluate this multiparameter nonlinear inversion algorithm, we must perform numerous simulations of larger and more realistic poroelastic inverse problems.

## Refereed

D.F. Aldridge, N.P. Symons, and L.C. Bartel, “Poroelastic Wave Propagation with a Velocity-Stress-Pressure Algorithm,” presented and in *Proceedings of the Third Biotechnology Conference on Poromechanics: Poromechanics III Biotechnology Centennial (1905–2005)*, Norman, Oklahoma, May 2005, vol. 1, pp. 253–258.

K. T. Kim, D.F. Aldridge, and N.P. Symons, “Seismic Wave Propagation in 3D Randomly-Heterogeneous Elastic Media,” presented and in *Proceedings of Expanded Abstracts, 75th Annual Meeting and International Exposition, Society of Exploration Geophysicists*, Houston, TX, November 2005, CD-ROM.

D.F. Aldridge, L.C. Bartel, and N.P. Symons, “Velocity-Stress-Pressure Algorithm for 3D Poroelastic Wave Propagation,” presented and in *Proceedings of Expanded Abstracts, 74th Annual Meeting and International Exposition, Society of Exploration Geophysicists*, Denver, CO, October 2004, vol. 1, pp. 1913–1920.

## Other Communications

D.F. Aldridge, N.P. Symons, and L.C. Bartel, “Poroelastic Wave Propagation with a 3D Velocity-Stress-Pressure Finite-Difference Algorithm,” presented and in *Proceedings of Abstracts, American Geophysical Union Annual Fall Meeting*, San Francisco, CA, December 2004, CD-ROM.

# Reverse-Time Seismic and Acoustic Wave Propagation: High Fidelity Subsurface Imaging and Location of Energy Sources

*79750*

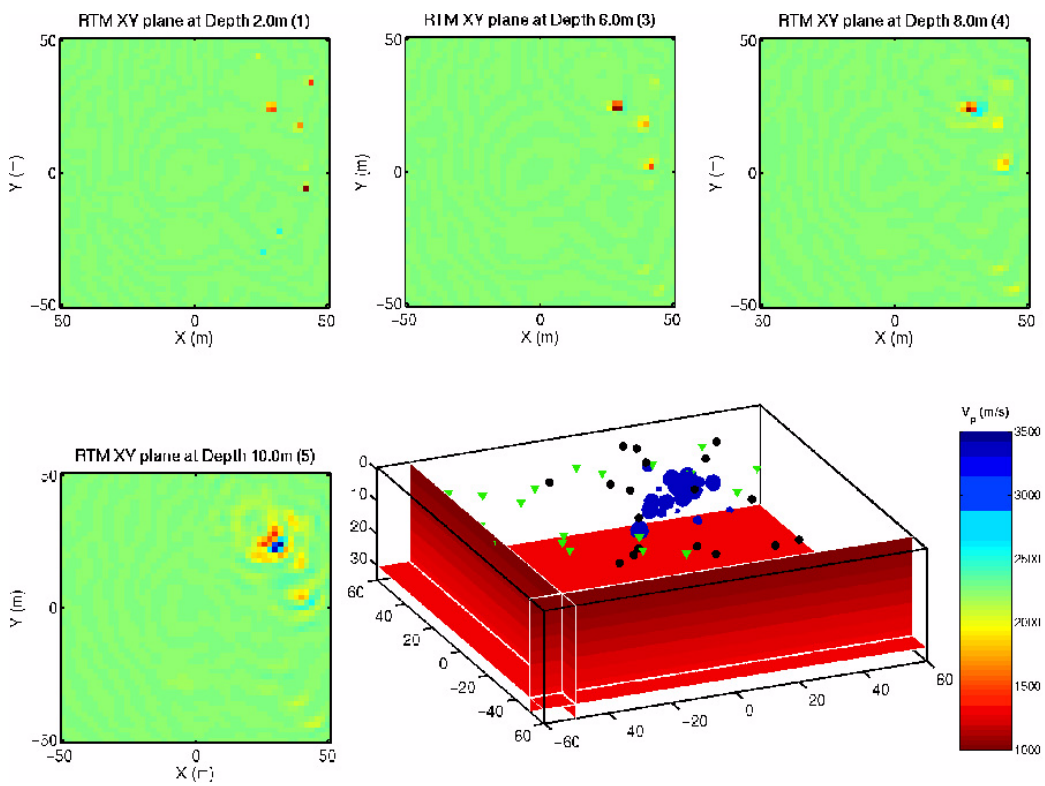
*L. C. Bartel, M. M. Haney, A. B. Doser, N. P. Symons, S. J. Younghouse, D. F. Aldridge, C. C. Ober*


## Abstract

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, and 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. Preliminary work using three-dimensional (3-D) seismic propagation codes demonstrates that when the correct source function is used, the time-reversed responses will vanish at the source location at t = 0, pinpointing the source location and excitation function. For unknown sources, RTM provides a method to identify the source waveform. RTM received some attention from the petroleum industry for improved imaging of the subsurface, but much research remains to develop RTM for 3-D elastic and/or anelastic media. Research issues include accurate amplitude recovery and imaging using a limited number of receivers, and developing a robust imaging condition, particularly for surface waves.


## Accomplishments

We completed the modification on our existing 3-D finite-difference codes for the RTM process. We found that it made more technical sense to include spherical spreading correction in the implantation of the imaging condition, and we completed the implementation of the excitation time imaging condition where we included




*Example of imaging a near surface boulder field using surface waves.*

spherical spreading corrections. As part of this work, we derived the analytic expressions for the imaging condition for source identification, imaging geologic structure, and imaging point scatterers. We completed the direct arrival removal methodology. We developed two signal processing methods for direct arrival removal: maximum likelihood with cross correlation, and eigenvector filter. We demonstrated surface wave implementation of the RTM method for imaging a near surface boulder field using simulated data. We are still investigating signal processing methods for separating the direct arrival from the forward scattered arrival.

Through an approximate mathematical approach to evaluating the migrated image over a single dipping reflector, we gained insight into the waveform results from RTM. We are able to make judgments on which of the two popular imaging conditions, cross-correlation or deconvolution, has the most desirable properties in terms of the image it produces. Future work will focus on sampling issues and the performance of the algorithm in imaging a more complex subsurface. This initial understanding of RTM should enable quantitative interpretation of the migrated amplitudes from field data. To be able to produce rapid forward and RTM solutions, we implemented a whole space Green's function solution, where we included the ability to calculate the responses from point scatterers with the Born approximation. This allows us to rapidly compute the direct arrivals, compute responses from point scatterers, and test various imaging conditions. Our work demonstrated that in a reflection survey, the direct arrivals can be removed from the data traces using an eigenvector filter method. In this filter approach, eigenvectors associated with the largest eigenvalues are related to the direct arrivals, while the eigenvectors associated with smaller eigenvalues correspond to the scattered responses. The signals are reconstructed from the appropriate eigenvectors. Under certain circumstances, we demonstrated that we can use an eigenvector decomposition method to produce the image of a scattering center when the cross-correlation and/or deconvolution image is formed without the direct arrival removal. When an image is produced by this eigenvector decomposition method, the image constructed from a particular eigenvector corresponds to the image of a particular scatterer.

## Refereed

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?" to be published in *Society of Exploration Geophysicists Expanded Abstracts*.

# Multi-Spectral Detection of Microfluidic Separation Products

*79751*

*C. C. Hayden, A. K. Luong, C. C. Gradinaru, A. K. Singh, D. W. Chandler*

## Abstract

With the technology for microanalytical devices pushing towards smaller sample volumes and concentrations, there is an increasing need for simultaneous detection of multiple biomolecules (pathogens, toxins, or biomarkers such as cytokines) at very low concentrations. To this end, we 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 will combine microfluidic separation techniques with our lifetime- and frequency-resolved detection capability to create a novel platform for unique scientific studies that will establish a differentiating scientific capability for Sandia.

In a single instrument, fluorescently tagged chemical complexes will be isolated by 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 and aggregation. 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 aim to use this approach to develop a new generation of detection capabilities for microfluidic separation systems to detect bioagents or biomarkers of a disease with unprecedented sensitivity (2–3 orders more sensitive than traditional fluorescence detectors) and multiplexing capability. This rapid screening tool has the potential to bring single molecule sensitivity to the separation process, while the multi-spectral detection will enable the simultaneous measurement of multiple analytes by using multiple fluorophores to selectively label species of interest. This approach could potentially reduce the possibility of false positive detection. Furthermore, the time-resolved detection will allow monitoring of binding events, such as binding of a toxin to an antibody, allowing development of diagnostic assays.

## Accomplishments

The objectives of this work are to develop a unique scientific tool for studies of chemical processes at the single molecule level, and to provide enhanced capabilities for multiplexed, ultra-sensitive separations and immunoassays. We are pursuing this goal by combining Sandia's expertise in micro-fluidic separations with our newly developed technology for spectrally- and temporally-resolved detection of single molecules. As a new scientific tool, the spectrally- and temporally-resolved single molecule fluorescence detection in microfluidic systems enables completely new approaches to fluorescence correlation spectroscopy, a widely used method for studying a variety of chemical processes in liquids, such as mobility and aggregation. 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 apparatus. The microfluidic technology offers unprecedented control of the chemical environment and flow conditions and affords the unique opportunity to study biomolecules without immobilization.

We demonstrated the capability for detecting single fluorophores freely diffusing in liquid samples. We made the first measurements with functionalized, water soluble quantum dots. We observed sharp spikes in fluorescence intensity when single quantum dots drifted into the focus of the excitation laser in our apparatus. The durations of these intensity spikes are typically a few milliseconds. We obtained fluorescence spectra and decays with excellent signal-to-noise ratios from single quantum dots observed for these short times. We used fluctuation correlation analysis of the signals from many quantum dots to extract an ~4 millisecond time scale for the diffusion of a quantum dot through the ~300 nm laser focal region. We made similar measurements on organic dye molecules, typically used for labeling biological molecules, and on small fluorophore labeled proteins. Correlation analysis on the fluorescence from the free dye molecules yields an ~200 microsecond time scale for the diffusion of the dye molecules through the laser focus. The smaller dye

molecules diffuse much faster than the quantum dots. These measurements in < 50 pM concentration aqueous dye solutions also contain significant contributions due to Raman scattering by water. The Raman scattering is readily identified by its very short (measurement limited) correlation time. Because our measurements are spectrally resolved, we can easily remove Raman components in the data analysis after acquisition, improving the signal-to-noise for the fluorophore measurements. These results demonstrate the necessary sensitivity for further experiments.

The development of time-stamping electronics and software that record the absolute arrival time of each photon detected in the apparatus enable these experiments. Our detection system is based on custom electronics that determine the wavelength and photon arrival time relative to the exciting laser pulse for each detected fluorescence photon. In addition, we are now able to stamp the information from each individual photon with its absolute arrival time (time-stamped) with an accuracy of 50 nanoseconds. This capability is crucial for studies that make use of photon arrival time statistics, such as correlation analysis.

The detection and analysis of fluorescence resonance energy transfer (FRET) signals provides important information for experiments designed to study binding processes of larger molecules. The measurement of FRET gives an indication of the distance between fluorophores on a 5 nm scale, well inside the resolution of optical microscopy. Thus, FRET can distinguish whether molecules are bound rather than just loosely associated. With carefully selected labeling, it can yield structural information on complexes. We recently demonstrated the capability of our apparatus to measure FRET between single fluorophore pairs (spFRET). In these experiments, we detect the complete spectrum of a single FRET pair showing the donor and acceptor contributions. The fluorescence decay as a function of wavelength is simultaneously determined. To our knowledge, this is the first measurement of the complete, correlated spectrum and decay associated with a spFRET process.

## Other Communications

C.C. Hayden, A.K. Luong, C.C. Gradinaru, and D.W. Chandler, “Time-Resolved Multi-Spectral Single Molecule Spectroscopy,” presented at the 53rd Annual Western Spectroscopy Association Conference, Pacific Grove, CA, February 2006.

# Atomic Force Microscopy Measurements of Adhesive Forces for Interpreting Colloid Transport

*90502*

*M. D. Reno, M. Piech*


## Abstract

The purpose of this project was to demonstrate the capability to directly measure interaction forces between carboxylate modified polystyrene microspheres (colloids) and common geologic substrates using the atomic force microscope (AFM) in conjunction with the colloid probe technique. We took measurements, while systematically varying experimental parameters (particle size, geologic substrate, ionic strength, and pH), with the intention of proving, by direct physical measurement, that colloidal particles are subject to entrapment in a secondary energy minimum. We attached microspheres with nominal diameters of 1.0, 2.0, and 3.2 microns to tipless silicon nitride cantilevers, creating a colloid probe. We prepared samples of common geologic substrates (quartz, feldspar, and mica) for use in a specialty AFM fluid cell. We administered potassium chloride solutions of varying ionic strength at a constant pH of 6 or 10 through the flow cell. We can use force distance curves generated from the AFM output data to identify the presence and magnitude of the secondary energy minimum.


## Accomplishments

The goals of this project were: (1) to determine appropriate colloids and substrate surfaces for use in experiments, (2) to develop a method for epoxying colloids to the atomic force microscope (AFM) cantilever, (3) to measure interaction forces between a colloid and geologic substrate, and (4) to compare force distance curves generated from AFM measurements to those predicted by Derjaguin Landau Verwey Overbeek (DLVO) theory. We achieved all goals.

We selected carboxylate modified polystyrene microspheres as the appropriate colloidal particle to employ because they are generally used in colloid transport studies as a conservative proxy. We chose the sizes of spheres employed in this work based on the need for a colloidal particle ($10^{-8}$ to $10^{-5}$ meters) combined with the need for a particle that could be attached to the tipless AFM cantilever. These particles are also attractive because they have well defined physical and chemical properties. Geologic substrates include pristine quartz, feldspar, and mica discs, approximately 1 centimeter in diameter. We chose these surfaces because of their common geologic occurrence and because they have already been used in several transport experiments.

We made several measurements in which colloid size, geologic substrate, and ionic strength of a KCl solution varied individually. We held the pH of the solution constant at 6 or 10. We devised these measurements based on recent research that identified a secondary energy minimum in effect for colloid deposition processes in porous media, a phenomenon that could explain the inadequate predictions of several colloid transport models to date. Other researchers predicted this secondary energy minima using DLVO theory and supported these calculations using results from column experiments; however, this minima has not yet been physically measured. We can use deflection distance data collected using the AFM to identify the presence and magnitude of the secondary energy minimum.

We developed and continue to improve an analytical technique with applications to both the research community active in studying sites for deep geologic isolation of radioactive waste, and the water security community. For example, in FY 2006, AFM measurements will be used to support the development of an algorithm ultimately intended to provide a real time operation tool for the decontamination of water distribution networks. Specifically, the AFM will be used to measure interaction forces between biofilms and the particles/surfaces with which they interact. These data will then be used as input to a molecular model that seeks to evaluate the effects of biofilms on decontamination of contaminated drinking water distribution systems.

# Computational and Information Sciences

## A Parallel Circuit Simulator for Cell Biology

*52541*
*R. L. Schiek, E. E. May, S. A. Hutchinson*

### Abstract

The next revolution in biology, building on the deluge of available genomics and proteomics data, will be based on understanding the genetic and metabolic signaling pathways that define how a cell, and ultimately an organism, works. Recent work shows that the mechanisms of gene expression and the interdependence of signaling pathways in the cell can be modeled as complex electrical circuits. Cells are essentially signal processors that receive inputs (deoxyribonucleic acid [DNA] sequences or genes), process information (transcription, for example), and produce outputs (proteins).

We are extending the Xyce circuit simulation program to create a large scale, parallel biocircuit simulator. This requires the development of individual circuit elements representing molecular machines, and an accurate description of the coupling between elements. Synthesis will continue up the functional hierarchy to the construction of single cells and the simulation of cell cultures. Unique to this simulation approach is the ability to simulate collections of cells interacting and influencing collective development. Initially, we will use the tool to model cell-level responses to environmental stresses. Ultimately, we will use Xyce's parallel capabilities to simulate a full cell culture.

Although computationally and biologically challenging to create, such a tool will aid the fundamental understanding of cellular biology. By providing a clearer understanding of how genetic networks influence cellular and cell culture level behavior, this work supports the further development of models for microbial and cellular processes of interest to the Department of Energy, such as for waste remediation and energy production.

### Accomplishments

We successfully completed a whole cell simulation of *Escherichia coli* central metabolism. The circuit netlist represents all metabolic and associated gene regulation networks for central metabolism. By setting the initial concentration of glucose to a large value relative to other metabolites in the system, we can simulate *E. coli* growth and biomass production on glucose medium for 10 hours of simulation time. This work compares well to experimental data previously reported in the literature. Additionally, we can couple our whole cell model to existing optimization tools, such as DAKOTA, to determine reaction rate parameters that result in simulation growth curves that are consistent with experimentally produced growth curves.

Further, we simulated groups of cells interacting and altering their development due to interactions with neighboring cells. As a test case, we studied a collection of 100 embryonic *Drosophila* cells connected through a diffusion limited environment. We allowed the system to differentiate normally with and without external stress placed on the cells. To address the many unknown parameters within the system, we coupled DAKOTA to Xyce to automate parameter sweeps and sensitivity analysis. We found that as the external system stress (in this case, noise in the initial concentration fields of control proteins) increased, the system's stability quickly diminished. Such a result implies that the natural system is either noise free or that there are

other error correction mechanisms in this tissue development system. Regardless, this represents a new technique to assess the dynamic stability of a proposed biological network even when the network crosses cell boundaries.

To aid in development and debugging of biological circuits, we developed a Java-based visualization tool, XMAP (Xyce Metabolic Analysis Program), for visualizing and interpreting the output data produced by Xyce for biological system simulation. This tool allows potential users to graphically view the metabolic and genetic state of whole cell and multi-cell systems. The dynamical behavior of the system is captured and can be viewed at various simulation levels. This tool will ultimately facilitate the use of the parallel circuit simulation tool by nonengineers.

## Refereed

R.L. Schiek and E.E. May, "Using Large Scale, Multi-Cellular Pathway Modeling to Understand Cellular Differentiation," presented and in *Proceedings of the American Institute of Chemical Engineers National Meeting*, Austin, TX, November 2004, vol. 5, CD-ROM.

R.L. Schiek and E.E. May, "Examining Tissue Differentiation Stability Through Large Scale, Multi-Cellular Pathway Modeling," presented and in *Proceedings of the NSTI Nanotech Technical Conference*, Anaheim, CA, May 2005, vol. 1, pp. 516–519.

E.E. May and R.L. Schiek, "Modeling Cellular Development Within a Circuit Context," presented at the SIAM Annual Conference, New Orleans, LA, July 2005.

## Other Communications

R.L. Schiek and E.E. May, "Using Circuit Modeling to Simulate Large Scale, Multi-Cellular, Biological Pathways," presented at the Numerical Aspects of Circuit and Device Modeling Workshop, Santa Fe, NM, June 2005.

E.E. May and R.L. Schiek, "A Parallel Large-Scale Network Simulator for Cell Biology," presented at the CIS External Technical Review, Albuquerque, NM, August 2005.

# Developing a Computationally Efficient Dynamic Multilevel Hybrid Optimization Scheme Using Multifidelity Model Interactions

*52542*

*J. P. Castro, A. A. Giunta, G. A. Gray, P. D. Hough*

## Abstract

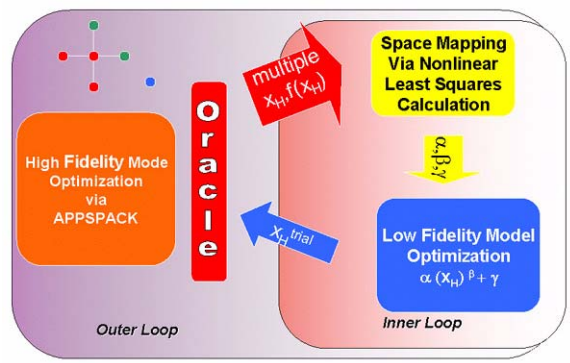
With the advent of massively parallel computers, computational speed has increased tremendously. Unfortunately, the comparable scaling of numerical and model complexity in combination with overburdened machines can still result in long compute times. This problem escalates when solving an optimization problem that requires many of these computationally intensive jobs to complete. Therefore, an algorithmic approach is proposed to make the optimization calculation more computationally efficient for a wide range of applications, including earth penetrator analysis, groundwater remediation, and electrical device modeling.

The goals of this project are to develop dynamic and computational time saving optimization algorithms by taking advantage of interactions between multifidelity models. If a system has models of different fidelities, the low fidelity, computationally cheaper model's design space may be used to drive the optimization. Within this design space, a set of rules or guidelines may determine the best optimization procedure. The low fidelity design space may then be mapped to the high fidelity design space to determine the optimum value, thus reducing the total computational time. This may be taken a step further by developing algorithms with feedback mechanisms so that the processes of determining the optimization scheme and model fidelity are done dynamically (this will be termed dynamic multifidelity-multilevel hybrid optimization).

This project addresses two primary technical challenges. First, we must develop algorithms and mechanisms for the dynamic multifidelity-multilevel hybrid optimization scheme. Second, we must integrate the newly developed schemes into software and link these to DAKOTA. The primary goals for FY 2005 were to develop a dynamic optimization scheme for an N-fidelity level of models and M-optimization strategies that allows for an unequal number of design variables between models of differing fidelity, implementation of an oracle, generalization of the space mapping approach, and expansion of current optimization benchmark calculations with new physics applications.

## Accomplishments

The goals for FY 2005 were: (1) to develop a limited dynamic optimization scheme for an N-fidelity level of models and M-optimization strategies that allows for an unequal number of design variables between models of differing fidelity, (2) to implement an oracle within DAKOTA/APPSPACK, (3) to generalize the space mapping approach, and (4) to expand upon current optimization benchmark calculations and metrics with new physics applications.




*APPS/space mapping scheme.*

The first three goals are all encapsulated within designing a multifidelity optimization strategy, accomplished through the completion of the following tasks:

(1) We developed an optimization scheme that incorporates a space mapping technique and the APPS (asynchronous parallel pattern search) method. It employs APPS to search for the optimum of the high fidelity model and simultaneously makes strategic calls to an inner loop that optimizes over the low fidelity model. The steps of this entire procedure are:

(a) Start the outer loop.
  (i) Optimize the high fidelity model, f_H, using the APPS algorithm.
  (ii) While optimizing, collect a set of N pairs (x_H, f_H).

(b) Start the inner loop.
  (i) Using the N high response pairs collected by the outer loop, obtain the space mapping

parameters via a least squares calculation to obtain a space mapping, P(x_H).

(ii) Optimize the low fidelity model within the space mapped high fidelity space by minimizing f_L(P(x_H)) with respect to x_H; obtain x_H*.

(c) Return x_H* to the APPS algorithm and determine if it is a new best point.

There are no limitations on the number of models or the number of design variables of each model.

(2) We incorporated our optimization scheme (inner loop) within the DAKOTA/APPSPACK software via an oracle. Oracles are used to predict a point at which a decrease in the objective function may be observed.

(3) We developed a generic space mapping implementation to account for an unequal number of design variables between high and low fidelity models. This implementation allows the capture of cross-dependencies between design variables by using a matrix like implementation of the space mapping function.

(4) The inner loop is executed using a generic series of scripts and template files that can be easily customized for specific applications. These scripts allow a very general form of mapping between the low and high fidelity models, including: any number of mapping parameters and design variables, syntax of mapping parameters and design variables, optimization techniques, functional form of the mapping, and number of responses required for mapping.

To test the APPS/space mapping strategy, we developed a set of test calculations using polynomial functions, Rosenbrock functions, and multivariable Rosenbrock functions. We used these test cases to thoroughly test the space mapping algorithm and, in particular, the case for nonequal design variables.

We applied our scheme to a groundwater remediation physics application. Remediation is a technique that is employed to reduce or minimize the negative. We demonstrated that the multifidelity optimization approach is a comparable method and thus may be a reasonable method for the solution of groundwater remediation problems.

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G.A. Gray, J.P. Castro, P.D. Hough, and A.A. Giunta, “Multifidelity Optimization Using Asynchronous Parallel Pattern Search and Space Mapping Techniques,” presented at the Institute for Mathematics and its Applications (IMA) Workshop: Career Options for Women in Mathematical Sciences, Minneapolis, MN, February 2005.

G.A. Gray, J.P. Castro, A.A. Giunta, and P.D. Hough, “Multifidelity Optimization via Pattern Search and Space Mapping,” presented at the 2005 SIAM Conference in the Geosciences, Avignon, France, June 2005.

A.A. Giunta, J.P. Castro, P.D. Hough, G.A. Gray, and M.S. Eldred, “Multifidelity Modeling Approaches in Simulation-Based Optimization,” presented at the 8th SIAM Conference on Optimization, Stockholm, Sweden, May 2005.

J.P. Castro, G.A. Gray, A.A. Giunta, P.D. Hough, and P.N. Demmie, “A New Scheme for Multifidelity Optimization Incorporating Pattern Search and Space Mapping,” presented at the 2005 SIAM Conference on Computational Science & Engineering, Orlando, FL, February 2005.

# Robust Large Scale Parallel Nonlinear Solvers for Simulations

*52543*

*T. G. Kolda, B. W. Bader*

## Abstract

Sandia has a wide range of highly nonlinear phenomena models that critically depend on robust nonlinear solvers. For example, the radiation-hardened Pentium processor simulation involves over 70,000 devices, and current solution methods are not robust because convergence depends on correctly setting parameters that are extremely problem dependent. Similar robustness issues occur in reacting flow simulations for the design of chemical vapor deposition reactors. Future goals, such as predicting the dispersion of toxic chemicals in water systems or buildings and modeling weapon electrical systems in hostile environments, will be even more difficult and will require advances in solver technology.

Our goal is to develop novel nonlinear solution methods that are both more robust and more efficient than Newton's method. We propose research on two complementary approaches to address this goal: the limited-memory Broyden's method and the Tensor method. Both methods focus on using an entirely different model rather than improving Newton's method directly. Broyden's method replaces the Jacobian with an approximation. Tensor methods are based on a higher order model and have been shown to overcome problems with singularity in the Jacobian.

## Accomplishments

We accomplished the following this year:

(1) We released the final versions of the Tensor method and Broyden's method in NOX. Release 7.0 of Trilinos will include NOX with the final production versions.

(2) We tested the Broyden method in the Charon code. Results obtained were similar to our previous evaluations in MPSalsa. We found the Broyden method at best to be ~10% faster in execution time than Newton's method. As the nonlinearity in the problem increased, the Broyden method required many more iterations to reach convergence. We recommend that Broyden's method be used on mildly nonlinear problems for increasing performance.

(3) We extended the Tensor algorithms to bifurcation tracking algorithms. In particular, we demonstrated those on the pitchfork tracking algorithm in the LOCA library. The current bordering algorithm required solves of near-singular systems. As the solution became more accurate, the Jacobian became more ill conditioned and started to cause convergence failures in the linear solver. Therefore, there was a limit on how accurate a solution we could obtain. The new algorithm generates a modified Jacobian that is always well-conditioned, thus allowing a tighter tolerance and more accurate answer. We demonstrated the algorithm on the Bratu problem in Matlab.

## Other Communications

B.W. Bader, R.P. Pawlowski, and T. Gibson-Kolda, "Robust Large-Scale Parallel Nonlinear Solvers for Simulations," Sandia Report SAND2005-6864, Albuquerque, NM, 2005.

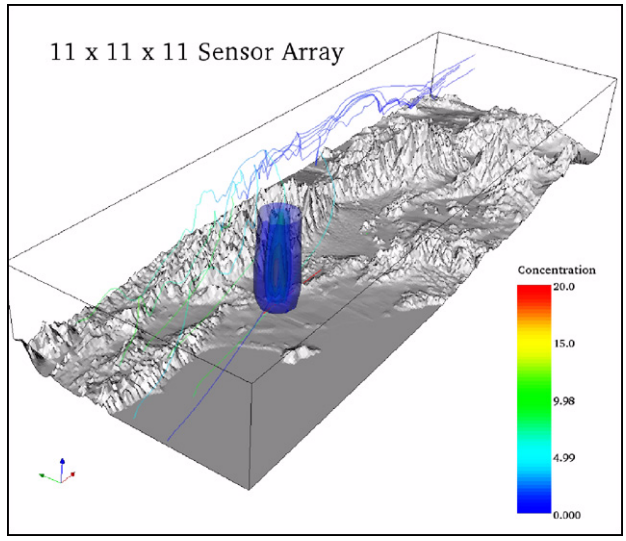
# Development of Computational Algorithms and Inversion Capabilities for Transport/Reaction Simulations of Chemical/Biological/Radiological Terrorist Attack Scenarios in Support of Homeland Security

*52544*

*B. G. van Bloemen Waanders, P. T. Boggs, K. R. Long, S. A. McKenna, R. A. Bartlett, J. N. Shadid, C. A. Phillips, W. E. Hart*

## Abstract

Our nation's critical infrastructure is extremely vulnerable to chemical/biological/radiological (CBR) terrorist attacks. Our goal was to develop countermeasure tools using numerical transport and optimization algorithms for urban canyons, internal facilities, and water distribution networks. In particular, we focused on developing methods to reconstruct attack events, control fluid flow in internal facilities, strategically place sensors, optimally design the decontamination process, generate appropriate boundary conditions for water distribution systems, and develop a risk assessment framework. We developed efficient multilevel solvers to simulate a high fidelity airport terminal model predicting chemical transport. We applied optimization algorithms to reconstruct initial conditions for fictitious CBR attacks. Most importantly, we uniquely applied these optimization algorithms to achieve real time performance for internal facilities, external flows, and water distribution systems. We developed a demand generator to examine the effect of temporal discretization of demand on flow and transport in a water distribution network. Finally, we developed combinatorial algorithms for sensor placement to consider a range of objective functions and formulations in addition to multicriteria objectives and the inclusion of uncertainty. We found that forcing optimality in one criterion usually resulted in poor performance for other metrics, but there exist solutions that effectively compromise across all objectives. Although we achieved real time performance by leveraging intrusive optimization methods, for certain domains the high computational demands still far exceeded our ideal goal of deploying this technology in an operational setting using a single processor computational platform. Consequently, we developed new reduced order modeling techniques for both the forward and the optimization problem. We developed risk assessment techniques for water distribution systems, and we applied them to a real dataset. As a result of this work, we received additional funding from the Department of Homeland Security and the Environmental Protection Agency for related projects.




*Contour of contaminant in the Los Angeles basin.*

## Accomplishments

We achieved all goals and milestones for this project. We developed real time source inversion techniques for water distribution systems and tested them on real large scale datasets. We developed special mixed integer quadratic programming methods to refine the solution from the regularized source inversion problem. We developed a subdomain method to address the computational efficiency and the ability to potentially invert for multiple attacks in parallel.

We developed and tested mixed integer optimization techniques on a range of datasets. We considered different formulations in addition to criteria, cost effects, temporal characterizations, and the effects of uncertainty. Furthermore, we addressed uncertainty by considering robust optimization methods.

Consumer demands are important source terms that need to be accurately characterized so that the underlying simulation of the optimization techniques can be as accurate as possible. We developed special Poisson

rectangular pulse techniques based on large sets of field data. We used these techniques and tested the variability of demands on the source inversion problem with surprisingly robust results. The source inversion did not appear to be affected as long as an adequate number of observation points were available.

The accuracy of the standard network simulator was evaluated and compared to high fidelity simulations and results. We concluded that the mixing rule is violated and incorrect chemical transport results at a spatial scale that consists of several junctions.

We applied risk assessment techniques, including Markov latent effect methods, to real datasets. This framework provides the ability to assess the possibilities of vulnerability given a characterization of operating conditions and possibilities associated with attack details.

For the air security problem, we were able to demonstrate high fidelity numerical simulation for chemical transport using efficient solver technologies. We demonstrated large scale parallel calculations of 100,000 to 100,000,000 unknowns using from 10 to 1000 processors. These calculations consisted of transient Navier-Stokes with large eddy simulation models.

We demonstrated real time source inversion for a steady state simulation of a three-dimensional airport model by separating off-line and on-line calculations. We also developed a near real time capability to perform source inversion for a contamination event in a large outdoor domain, such as the Los Angeles Basin. The computational requirements are still large for this problem, and so we designed reduced order techniques for both the forward and the optimization problem.

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B. Bader, O. Ghattas, B. van Bloemen Waanders, and K. Willcox, “An Optimization Framework for Goal-Oriented, Model-Based Reduction of Large-Scale Systems,” presented and in *Proceedings of the Joint 44th IEEE CDC and ECC Conference*, Seville, Spain, December 2005, CD-ROM.

V. Akcelik, G. Biros, A. Draganescu, J. Hill, O. Ghattas, and B. van Bloemen Waanders, “Dynamic Data-Driven Inversion for Terascale Simulations: Real-Time Identification of Airborne Contaminants,” presented and in *Proceedings of SC05*, Seattle, WA, November 2005, CD-ROM.

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C.D. Laird, L.T. Biegler, B.G. van Bloemen Waanders, and R.A. Bartlett, “Contamination Source Determination for Water Networks,” *Journal of Water Resources Planning and Management*, vol. 131, p. 125, 2005.

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B. van Bloemen Waanders, G. Hammond, J. Shadid, S.S. Collis, and R. Murary, “A Comparison of Navier-Stokes and Network Models to Predict Chemical Transport in Municipal Water Distribution Systems,” presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

S.A. McKenna, B.G. van Bloemen Waanders, C.D. Laird, S.G. Buchberger, Z. Li, and R. Janke, “Source Location Inversion and the Effect of Stochastically Varying Demand,” presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

C.D. Laird, L.T. Biegler, and B.G. van Bloemen Waanders, “A Mixed Integer Approach for Obtaining Unique Solutions in Source Inversion of Drinking Water Networks presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

J.W. Berry, W.E. Hart, C.A. Phillips, J.G. Uber, and J.-P. Watson, “Validation and Assessment of Integer Programming Sensor Placement Models,” presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

J.W. Berry, W.E. Hart, and C.A. Phillips, "Scalability of Integer Programming Computations for Sensor Placement in Municipal Water Networks," presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

J.W. Berry, W.E. Hart, C.A. Phillips, J.G. Uber, and J.-P. Watson, "Validation and Assessment of Integer Programming Sensor Placement Models," presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

J.W. Berry, W.E. Hart, C.A. Phillips, J.G. Uber, and T.M. Walski, "Water Quality Sensor Placement in Water Networks with Budget Constraints," presented and in *Proceedings of the World Water & Environmental Resources Congress*, Anchorage, AK, May 2005, vol. 1, CD-ROM.

# A Method of Evaluating Research Using New Innovation, Risk, and Impact Indicators

*59916*

*K. W. Boyack, G. S. Davidson*

## Abstract

In this age of modern information availability, organizations are searching for ways to capture tacit knowledge and use that knowledge to evaluate and select between new ideas. We are continually falling behind in our retention of knowledge due to the rapidity with which new information becomes available and from a lack of good tools to help us retain what we learn. We propose a method of evaluating research ideas by creating new indicators that, in essence, provide a numerical history of accumulated knowledge. These are research community based innovation, risk, and impact indicators that can give the researcher, administrator, or evaluator a means to quickly evaluate an idea within the context of the history of similar ideas. These indicators will also facilitate focused access to pertinent literature for those with a desire to gain more detailed knowledge and thereby improve their ideas.

Large scale maps of science covering several years have been generated, and the maps are clustered to the research community level. A research community is a cluster of about 10 papers published on the same topic during a given year. Metrics, such as vitality, can be calculated for each community, both past and current. Queries can be made to find communities of relevance to a given topic. Higher ranking communities that are linked to those returned by a query can also be returned, thus leading researchers to potentially more vital areas in which to work. Potential applications of these data include evaluation of current research at aggregated levels (e.g., corporate, department, etc.) and suggestion of opportunities for collaboration. At the individual level, the results of this study can be used to guide future research into more vital areas.

## Accomplishments

Over the past several years, researchers at Sandia developed techniques for clustering very large segments of the technical literature using sources such as Thomson's Science Citation Index. The primary objective of this work was to develop indicators of potential impact at the paper level to enhance planning and evaluation of research. These indicators can also be aggregated at different levels to enable profiling of departments, institutions, agencies, etc. Results of this work are presented as maps of science and technology with various overlays corresponding to the indicators associated with a particular search or question.

Our maps of science and technology are generated using a multistep process and consist of two maps calculated at different levels. The first map is a structural map and shows the various disciplines in science and technology and their relationships to one another. The structural map provides an easily understandable visual picture of science and technology as a whole and is the template upon which query results can be presented. This map is generated using coupling coefficients between pairs of journals (or conference proceedings). Nodes on the map represent subdisciplines, and lines represent major coupling pathways between sets of journals.

The second map is generated from the individual papers themselves. This map consists of 997,775 papers (indexed in 2003) from Thomson's Science Citation Index, Social Sciences Citation Index, and ISI Proceedings databases. The papers are clustered using bibliographic coupling; two papers are coupled if they share common references. Papers that are strongly coupled end up in the same cluster and are generally focused on the same research topic. The 2003 map consists of 117,435 separate clusters of papers. A cluster of papers, or research community as we call it, is the unit of analysis that we use. For presentation purposes, each community is assigned to the appropriate journal cluster in the structural map. The structural map can thus display information related to research communities, aggregated to the subdiscipline level.

Our work focused on (1) demonstrating the accuracy of our mapping approaches at both the structural and detailed mapping levels, (2) generating the largest and most accurate maps of science ever created, and (3) generating explanatory metrics at the community level.

Our accomplishments include three papers detailing our processes and their accuracy. These are the most detailed mapping accuracy studies ever published. We also created maps for years 2001–2003 with

metrics and indicators. The maps are linked to provide continuity between years and to show the dynamic nature of communities over time. Results of this work have also been used in various projects at Sandia to provide research evaluation and to identify targets for collaborative opportunities.

## Refereed

R. Klavans and K.W. Boyack, "Mapping World-Wide Science at the Paper Level," presented and in *Proceedings of ISSI 2005, 10th International Conference of the International Society for Scientometrics and Informetrics*, Stockholm, Sweden, July 2005, vol. 1, pp. 426–436

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## Other Communications

K.W. Boyack, "Mapping World-Wide Science at the Paper Level," presented at the Regenstrief Institute, Indianapolis, IN, March 2005.

K.W. Boyack, "Use of Visualization Techniques to Guide the S&T Enterprise," presented at the EEG/SCIP CTI 2004, Boston, MA, October 2004.

K.W. Boyack, "Mapping All of Science and Technology at the Paper Level," presented at the American Association of Geographers, Denver, CO, April 2005.

# Massively Parallel Scalable Atmosphere Model

*67015*

*W. F. Spotz, M. B. Boslough, R. C. Schmidt, M. A. Taylor*

## Abstract

The purpose of this project is to develop a massively parallel, highly scalable, element based atmospheric dynamical core. A dynamical core is defined as that part of a weather or climate model that solves the primitive equations. These equations are the stratified Eulerian equations of fluid motion in spherical coordinates and constitute the most computationally intensive calculations in an atmosphere or ocean model. Currently, the world's fastest dynamical core runs on the Japanese Earth Simulator, which recently achieved over 26 teraflops on 5120 vector processors at an equatorial resolution of roughly 10 km. Our objective is to match this in absolute performance measures and surpass it in cost performance measures on the next generation of proposed Department of Energy (DOE) architectures.

The Japanese dynamical core uses the spectral transform method (STM), a common production-level dynamical core that has known scalability issues. Currently, the most scalable dynamical core is the Spectral Element Atmosphere Model (SEAM), a research code developed at the National Center for Atmospheric Research (NCAR), which won an honorable mention in the 2001 Gordon Bell competition.

Element based approaches permit new capabilities impossible for the STM, including both static mesh refinement around geographic features and dynamic refinement around moving fronts. This capability will make SEAM particularly viable for emergency response modeling of radiological, chemical, or biological dispersions. This will be made possible by tools developed at Sandia such as Aztec, Chaco, and Zoltan. Research will consist of exploring the scalability of SEAM on modern DOE architectures, of improving the time-stepping capability of SEAM, of exploring triangular spectral elements, of adding community standard subgrid physics parameterizations to SEAM, of stabilizing the resulting coupled dynamics/physics model, and of using the coupled dynamics/physics model to run standard inter-comparison tests.

## Accomplishments

In FY 2005, we added many additional capabilities to the SEAM code. Primary among these is that we upgraded semi-implicit time stepping from the two-dimensional (2-D) (shallow water) version to the 3-D (primitive equation) version. It now includes an eigenmode decomposition that greatly reduces the required iterations to solve a time step.

Semi-implicit to explicit performance comparison studies indicate a factor of 3 to 4 increase in integration rate (simulated days per wall-clock day) for small to moderate processor counts.

We obtained parallel performance and scalability results (for explicit time-stepping) on Red Storm and BlueGene/L. On Red Storm, we obtained very good scalability up to 7200 processors, which was the maximum we had access to. On BlueGene/L, we obtained good scalability up to approximately 25,000 processors, but saw poor scalability above that. On both machines, we were able to obtain a peak performance of roughly 4 teraflops (~20 days/day at 10 km resolution).

We exploited SEAM scalability to perform a 3-D mesh convergence study of the breakdown of the polar vortex. This allowed us to characterize which features converge at typical climate resolutions and which features require substantially higher resolution.

We started a collaboration with the Navy's spectral element forecast model development effort. We developed a novel and innovative numerical technique to extend the spectral element method from quadrilateral elements to triangular elements. The Navy recently used this method successfully in their 2-D research forecast code.

At a planning meeting in December with NCAR collaborators, we decided to prioritize the integration of SEAM with subgrid physics parameterizations, and decided that Sandia would assume more responsibilities for accomplishing this. Therefore, additional development work focused on integrating SEAM and the Community Atmosphere Model (CAM) subgrid physics parameterizations. Initial efforts resulted in instabilities that proved difficult to track down. We are currently working with a reduced and simplified set of physics parameterizations ("Emanuel physics") to help us isolate and correct these problems.

We spent limited time looking at improved turbulence and cloud-resolving models that could better take advantage of SEAM's capabilities. We are pursuing the idea of 1-D turbulence in a cloud super-parameterization.

## Refereed

J. Dennis, A. Fournier, W. 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.

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## Other Communications

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# High Performance Processing Architecture

*67016*

*K. D. Underwood, K. S. Hemmert*

## Abstract

Processor architecture is one aspect of supercomputer design that is typically unquestioned. Microprocessors are the basis of most major supercomputers. Even Blue Gene/L, which uses a somewhat novel approach to system design, is based on commodity embedded processor cores. As the barriers that traditional processors face mount, it is necessary to consider the implications of alternative processor architectures for scientific codes.

We are studying the impact of reconfigurable computing on supercomputers at the petaflops scale and beyond. Reconfigurable computing is defined as the use of programmable hardware to provide application specific acceleration. It is an alternative approach to processor architecture that is receiving significant attention in the research community; however, research is currently focused on application domains and system architectures that are very different from high end scientific computing at the petaflops scale. This project is providing insight into issues facing the integration of reconfigurable computing into future supercomputers. Specifically, we are studying the use of field-programmable gate arrays (FPGAs) to provide reconfigurable computing at the petaflops scale.

The approach is two pronged. One focus is on actual implementations of key computational operations associated with Sandia applications. Application implementation using FPGAs is a time consuming process; thus, it is impractical to implement a large number of applications as part of this effort. Instead, we are implementing a small set of core operations to investigate the characteristics of FPGAs and their interface to the system. We will use data from these implementations to support the second focus of this proposal: modeling of key Sandia applications. We will leverage architecture simulation along with data from initial implementations to gain understanding of FPGAs at the petaflops scale.

This year, we performed an in-depth study of the fast Fourier transform (FFT) operation. We also found two appropriate applications to simulate with the operations we studied. We also significantly enhanced the simulation environment this year.

## Accomplishments

There were three major objectives for FY 2005, and we met each of them. The first goal was to select a set of applications to model. We currently plan to model the large-scale atomic/molecular massively parallel simulator (LAMMPS) and the massively parallel quantum chemistry (MPQC) codes and are considering an additional code using a sparse solver. Each of these has an aspect that we believe can be enhanced with FPGAs. Our selection of the subset of these applications was guided by cooperation with our Sandia colleagues on the complementary LDRD Project 67018, “Enhancing Simulation Performance on Clusters with Configurable Auxiliary Devices.”

The second major objective was to implement a set of computational kernels in FPGAs. We implemented multiple variants of the FFT for FPGAs, and models of both sparse and dense linear solvers are in progress. Results from the work with the FFT led to further refinement of the floating-point units. Inquiries about potentially licensing this work increased, and we licensed the floating-point units under an open source license.

The final objectives for this year were to build three representative FPGA system architectures in the simulator and to perform parameter sweeps over those architectures. We specified three configurations and focused work with the simulator on much higher fidelity simulations rather than immediately jumping to parameter sweeps. The end result should allow us to achieve better answers next year.

## Refereed

K.S. Hemmert and K.D. Underwood, “An Analysis of the Double-Precision Floating-Point FFT on FPGAs,” presented and in *Proceedings of the IEEE Symposium on Field-Programmable Custom Computing Machines*, Napa, CA, April 2005, pp. 171–180.

# Substructured Multibody Molecular Dynamics

*67017*

*P. S. Crozier, M. J. Stevens, S. J. Plimpton, R. B. Lehoucq*

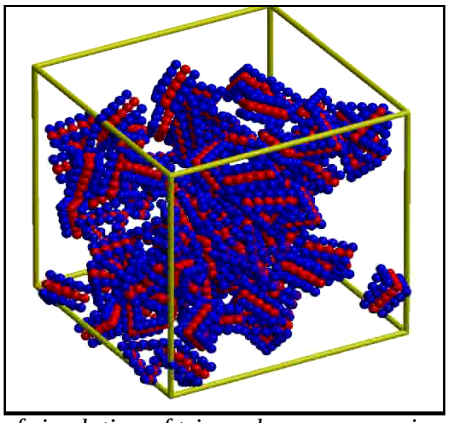
## Abstract

Over the past few decades, the molecular dynamics (MD) method of exploring molecular level structures, dynamics, and behavior has transformed from a computational novelty to an indispensable research tool. One of the major remaining hurdles facing MD is an inherent timescale limitation that restricts researchers to the nanosecond regime. The goals of this project focus on dramatically increasing the timescale range available to MD practitioners through intelligent elimination of non-essential degrees of freedom via rigidification and coarse graining and through improved multiple time-stepping, implicit solvent methods, automatic substructuring, and other MD algorithm improvements. Early project successes include major code development work on our LAMMPS (large-scale atomic/molecular massively parallel simulator) MD package, enabling rigid body dynamics and generalized multiple time-stepping. We also wrote code that doubles LAMMPS's speed on long range electrostatics simulations by tabulating expensive force calculations and using an efficient bitmapped lookup algorithm. The code also enables the use of highly efficient tabulated coarse grain force fields that are derived from atomistic force field simulations. Ongoing work focuses on improved implicit solvent methods, automatic substructuring, coupled rigid body simulations, and testing of the code on important computational molecular biology and nanoscience applications. This work focuses Sandia's impressive computational capabilities on the development of next generation molecular simulation tools that will help bring about the nanotechnology and biotechnology revolutions.

## Accomplishments

We made numerous changes, improvements, additions, and upgrades to the LAMMPS MD code, including improved rigid body dynamics, better multiple time-stepping, and enabling of multibillion atom simulations. Details of some of the code improvements are given below.

- *Improved rigid body dynamics*. We implemented and tested the Richardson algorithm and an iterative integration algorithm for rigid body dynamics, which yield much improved energy conservation as compared to the "vanilla" integration method originally implemented in LAMMPS's rigid body dynamics routines. We also made code modifications to correctly compute the temperature and pressure of a substructured, partly rigid system by accounting for degrees of freedom that are lost by rigidifying groups of atoms. We made additional improvements in the way pair and bond interactions are masked within rigid bodies to avoid unnecessary computational expense.



*Movie of simulation of triangular coarse-grain particles using the LAMMPS molecular simulation package.*

- *Lookup tables*. We made substantial speed and capability improvements to LAMMPS by adding lookup tables that accelerate coulombic interaction calculations and enable the use of radial, pair-wise tabulated force fields. Recent improvements in coarse grained force field fitting based on atomistic force field simulation opened the way to superior accuracy coarse-grained MD simulation of large-scale biomolecular and nanoscale systems. Additions to the LAMMPS source code make it possible to perform simulations using essentially any tabulated coarse grain potential. We included three table lookup options in the code, listed here in order of increasing accuracy per grid point and computational cost: truncation, linear interpolation, and cubic spline lookup. We also included bitmapping for faster lookup than type casting. Tabulated coulombic interactions calculated using the new algorithm are more accurate and roughly twice as fast as those calculated using the original algorithm.

- *Code changes enabling simulation of very large systems*. We ported LAMMPS to Lawrence Livermore National Laboratory's new IBM BG/L supercomputer and ran the LAMMPS benchmark suite on up to 65,536 processors and up to 4 x $10^{10}$ atoms. These runs are made possible by code changes that enable the simulation of billions of atoms by properly handling integer overflows in code for atom counts and thermodynamics and by replacing O(N) lookup arrays with O(N/P) hash tables.
- *Additional code improvements*. We implemented a generalized reversible reference system propagator algorithm in LAMMPS to allow multiple time-stepping on an arbitrary number of levels. Atomistic regions can be simulated on inner loops with the larger rigid bodies on outer loops, yielding substantial savings in computational cost. In conjunction with the Genomics:GTL program, we implemented the targeted molecular dynamics (TMD) method for accelerating large-scale biomolecular (or other) conformational transitions, and tested the code on simulations of RuBisCO gating. We also implemented class 2 force fields for multibody interaction force fields and made it possible to use multiple sets of pair or bond potentials within a single simulation so that hybrid bio/inorganic/metallic systems can be simulated. We also revamped our CHARMM-to-LAMMPS conversion script to facilitate the conversion of very large biomolecular systems into a format usable by LAMMPS. The improved script dramatically simplified the process and is available to LAMMPS users.
- *Applications*. We performed atomistic simulations of dioleoyl-3-trimethylammonium-propane, dioleoyl phosphatidylethanolamine, and dioleoylphosphatidylcholine lipid bilayers in preparation for coarse grained force field fitting based on the trajectories of the atomistic force field simulations. We tested and applied the TMD code in the simulation of the RuBisCO gating mechanism in conjunction with the Genomics:GTL program. Many of the code improvements completed as part of the project made these compute-resource-intensive RuBisCO simulations possible. We also performed all rigid body simulations of granular nanoparticles. These simulations allowed us to test the rigid body dynamics code improvements.

## Refereed

J.N. Sachs, P.S. Crozier, and T.B. Woolf, "Atomistic Simulations of Biologically Realistic Transmembrane Potential Gradients" *Journal of Chemical Physics*, vol. 121, pp. 10847–10851, December 2004.

# Enhancing Simulation Performance on Clusters with Configurable Auxiliary Devices

*67018*

*C. D. Ulmer, R. C. Armstrong, D. C. Thompson, M. L. Leininger*

## Abstract

Current generation high performance computing (HPC) systems rely almost exclusively on general purpose processors to perform the complex calculations required 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) are 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 FPGA capabilities present an opportunity to exploit these devices as economical "soft ASICs" that can accelerate scientific calculations.

In this project, we are addressing a critical challenge in RC: the integration of FPGA accelerators into HPC system architectures. Specifically, we are examining integration in two different contexts. First, in our network based approach to integration, we are developing FPGA hardware that enables us to connect FPGA resources to our existing HPC systems' interconnection networks. Second, in our host based approach, we are developing infrastructure to enable users to leverage FPGAs that are available in the local host architecture. We are analyzing the trade-offs associated with these approaches to determine which method provides the best coupling between host processors and FPGA accelerators.

As a means of demonstrating the benefits of this research, we are also porting computational kernels from scientific applications to our RC platforms. This effort leverages and complements the work being performed in the "High Performance Processing Architecture" effort, LDRD Project 67016, which is addressing the design of high precision floating-point units for FPGAs.

## Accomplishments

We made significant progress in multiple research areas:

(1) Refinement of Reliable Transmission Unit for a Gigabit Ethernet (GigE):

In order to investigate the network based form of system integration, we constructed communication hardware that enables an FPGA to interact with commodity network hardware and software. Specifically, we implemented a GigE unit and a transmission control protocol (TCP) unit for the Xilinx Virtex II/Pro FPGA. This year, we made the communication units more robust and hand tuned the protocol implementation to yield better performance.

(2) Network Transport Unit for Visualization:

In order to enable visualization research to be performed on FPGAs, we implemented a unit that converts graphics commands into a format that can be transported over the network. This unit utilizes a well known OpenGL programming interface and is capable of interacting with the Chromium standard for graphics transport. We implemented a functional prototype on a commodity FPGA card that uses our TCP/GigE units to transmit graphics commands over the network to a remote host for rendering.

(3) Network Intrusion Detection System (NIDS):

Based on our work with FPGA networks, we constructed an in-line NIDS that is capable of sanitizing multiple GigE links of malicious packets based on the Snort rule set.

(4) Initial Cray XD1 Investigation:

We examined the Cray XD1 computing system as an RC platform. The XD1 provides both general purpose central processing units (CPUs) and FPGA accelerators in an integrated system. We developed basic infrastructure to utilize these FPGAs and implemented multiple test applications to observe the strengths and weaknesses of this architecture.

(5) Isosurfacing Application:

We wrote an FPGA module for performing isosurfacing on three-dimensional data sets. This module performs a threshold calculation on all input values and then transforms the results into a collection of renderable triangles using the Marching Cubes algorithm. We tested the algorithm in simulation on a computed tomography (CT) data set, and implemented a prototype design using a commercial FPGA card.

(6) Early Compiler Analysis:

One of the hardships associated with hardware design is the high overhead associated with development. In order to decrease this cost, we constructed preliminary tools for automating the process of converting an algorithm to hardware. These tools construct a data-flow graph for an algorithm and then map the graph to synthesizable hardware. While these tools are experimental in nature, we believe they will be essential to our effort to adapt more algorithms to hardware.

(7) Triangle Intersection Hardware:

A common operation performed in visualization work is detecting whether a ray of light will hit a triangle or not. As a means of driving our compiler work, we chose to implement a triangle intersection algorithm in hardware using a fixed number of floating-point units. In this study, we examined the performance trade-offs associated with different approaches to implementing the data path.

(8) Linear Sorting Array:

Data sorting is a fundamental operation in computer science. As a means of demonstrating how hardware can be employed to increase performance, we constructed a linear sorting array for FPGAs that sorts data values in a vector from smallest to largest. Our implementation on the Cray XD1 provided three times the performance of the best software implementation, even though the FPGA had the additional burden of transferring the data into its own address space.

## Refereed

C.R. Clark and C.D. Ulmer, “Network Intrusion Detection Systems on FPGAs with On-Chip Network Interfaces,” presented and in *Proceedings of Applied Reconfigurable Computing*, Algarve, Portugal, February 2005, CD-ROM.

## Other Communications

C.D. Ulmer, R. Hilles, and D.C. Thompson, “Reconfigurable Computing Aspects of the CRAY XD1,” *Proceedings of the CRAY User Group*, Albuquerque, NM, May 2005, CD-ROM.

# Penetrator Reliability Investigation and Design Exploration (PRIDE)

*67020*

*M. L. Martinez-Canales, G. A. Gray, R. R. Settgast, P. D. Hough, T. G. Trucano, M. L. Chiesa, L. P. Swiler, R. Heaphy*

## Abstract

Earth penetrators are an important tool in our defense arsenal and are especially important weapons for defeating hardened deeply buried targets. However, designing penetrators to survive the extremely high loads resulting from penetration of hard geologies at high impact velocities is typically a difficult and sometimes impossible task. We will employ modeling and simulation (M&S) in the design process as an alternative to expensive hand designing and field tests. Because conventional M&S approaches are also cost prohibitive, very few attempts are made to computationally optimize the penetrator shape to reduce lateral loads and increase the probability of penetrator survival. The goal of this project is to reduce the computational expense of the overall design process to enable the design of credible, higher quality, reliable, and robust earth penetrators (EPs).

The focus of our work is research and algorithmic development in optimization under uncertainty (OUU) problems. There are three main challenges for this work. The first challenge requires leveraging small local samples, already needed by some optimization methods, to build effective surrogate models. We will compare trade-offs in quality and computational cost between surrogate models. The second challenge is to develop a methodical design process based on multi-resolution, multi-fidelity models. Using Gaussian processes (GPs) to model simulation responses, we will build reduced-order or surrogate models that are effective and computationally inexpensive. We will make a significant effort to understand mesh sensitivities, convergence issues, and design extrapolation errors in a multi-fidelity hierarchy. We will also develop and assess credibility metrics at each resolution level. The third challenge involves the development of tools that are computationally feasible and the implementation of design processes that are useful to Sandia and the weapons community. We will demonstrate these concepts on a prototype EP calculation on a high performance computing machine.

## Other Communications

## Accomplishments

A significant component of our second year efforts was analyzing statistically based parametric studies and the development of reduced-order models based on Bayesian approaches for EP design. From these parameter studies, we determined that the Pen-X nose tip length, penetration shaft cavity radius, penetrator impact velocity, and penetrator offset from the penetration shaft central axis all influenced earth penetration. We treated the penetrator tip length as a design variable; we treated the delivery and target variables as uncertain variables in our studies. We built regression models based on these variables for the ground displacement response and used them in our multi-fidelity Bayesian autoregressive process (MF-BAP) implementation. Initial results from MF-BAP show that we can use cheaper lower fidelity responses, together with a systematic model bias correction based on a GP emulator, to estimate mid-fidelity earth displacement responses (with a maximum 5% relative error). Using just the lower-fidelity model alone to predict mid-fidelity displacement, we obtain a maximum of 24% relative error on the parameter space tested. The lower fidelity model plus the systematic model bias correction constitute the reduced order model of the higher order models.

We implemented the MF-BAP/GP as a C++ object-oriented program. Efforts to incorporate the MF-BAP/GP, as a surrogate, into a trust-region-based OUU algorithm are under way. While this approach is mathematically sound, it requires derivative information that we must additionally compute. The need for potential additional computing requirements in this approach led us to start investigating a strategy for implementing a GP within a pattern-search (derivative-free) optimization method.

We are also considering decision metrics that will automate the decision of when higher fidelity information must be gathered, when lower fidelity information must be gathered, and when gathering additional lower fidelity information no longer improves the design process.

L. Swiler and T. Trucano, “Bayesian Methods in Engi-

neering Design Problems," Sandia Report SAND-2005-3294, Livermore, CA, 2005.

M. Martinez-Canales, R. Heaphy, P. Hough, M. Chiesa, R. Settgast, L. Swiler, and T. Trucano, "Penetrator Reliability Investigation and Design Exploration, Part 1: Low Fidelity Penetrator Design Studies," Sandia Report, Livermore, CA: Sandia National Laboratories, 2005.

# Topology Optimization for Improving Sensor Performance

*67021*

*K. R. Long, E. B. Cummings, B. G. van Bloemen Waanders, P. T. Boggs*

## Abstract

Optimal design of the geometry of a device occurs in many areas of engineering. In many problems, it is not sufficient to deform the shape of boundary surfaces; rather, we must vary the topology of the problem domain by introducing holes or islands. We introduced new algorithms and corresponding high performance software for topology optimization. Algorithmic work has concentrated on global optimization; we developed a multiscale global optimization strategy that is tailored to the geometric nature of this problem. We also developed a new formulation of level-set based topology optimization, which yields significant improvements in robustness and efficiency over both our own previous work and recent work by others. Furthermore, in order to improve the efficiency of local optimizations arising in this problem, we developed a system for adaptive choice of memory size in the limited memory (LM) BFGS (Broyden-Fletcher-Goldfarb-Shanno) optimization algorithm, which can result in a 30–50% reduction in the number of iterations required for convergence.

We also continued to improve the Sundance software package for large scale partial differential equation (PDE)-constrained optimization used throughout this work. We applied these new methods to two problems of significant interest in Sandia's homeland security efforts: the design of a minimum dispersion, microfluidic sample insertion device and the placement of sensors yielding the optimal recovery of a toxin source for a suite of attack scenarios in a model of turbulent flow in a large indoor area.

## Accomplishments

(1) Much of our algorithmic work has concentrated on developing a global optimization strategy that has proven efficient for topology optimization.

Our approach to global optimization was motivated by the tunneling method in which one begins with a local minimum, then picks a random direction in design space, and systematically searches it hoping to find an improvement in function value. In problems with many design variables and expensive function evaluations, we need to generate search directions with a high probability of success. We developed such a scheme tailored to the known geometric structure of our problem: rather than randomly perturbing the level-set function everywhere, we can advance a coarse grained approximation to the field by an amount chosen to move the level set randomly in space by a desired distance. Coarse-graining generates search directions in a subspace in which changes to the design variable produce macroscopically significant changes in the problem geometry.

We adopt an approach in which upwards steps have some probability of acceptance; as in simulated annealing, the probability of acceptance depends on the increase in objective function and can be made more unlikely as the simulation proceeds. Upon provisionally accepting a step, we then use it as the starting point of a new local optimization subproblem. In practice, we found that between 5% and 20% of the time, upwards steps led to an improved design. After several tens of iterations of this process, we were able to find a minimizer about which no further improvement could be found.

(2) In our work with high resolution models and global optimization, we discovered that it was fruitful to allow greater freedom for the thickness of the island boundary than allowed by the convolution mapping we previously developed, which restricts the transition in density from 0 to 1 to occur within a single element. A thesis by one of our team members introduced a level-set formulation allowing this freedom. However, the method in its original form proved unsuccessful when applied to our problems because of a non-convex slope penalty term. We improved the method by replacing slope penalization with an inequality constraint formulation, which proves to be both more efficient and more robust (in terms of fewer convergence failures) than previous convolution methods. Further, it is just as easily implemented in finite-element codes. All of our level set work now uses this formulation.

(3) Our global optimization strategy involves a sequence of local optimizations; it is, therefore, desirable to make these local optimizations as efficient as possible. In collaboration with Richard Byrd of the University of Colorado, we developed an improvement to the LM-BFGS algorithm used as our local optimizer.

The LM-BFGS method constructs an approximate Hessian from information obtained during several previous iterates. Its performance is sensitive to the "memory size," i.e., the number of iterates used; too few, and the Hessian is approximated too crudely; too many, and the information is too "old" to be useful in approximating the current behavior of the function. Researchers have developed a method for adaptively choosing memory size. For nonquadratic objective functions, the method typically results in a reduction of 30–50% in the number of LM-BFGS iterations required for convergence.

(4) With the global optimization strategy and reformulated level-set method described above, we produced a minimum dispersion microfluidic expanding channel with a 3:1 outlet:inlet ratio, having a final topology that is significantly different from the initial topology.

(5) We applied our topology optimization algorithms to the problem of placing sensors to provide optimal recovery of the location and intensity of a chemical attack in steady turbulent flow.

# A Mathematical Framework for Multiscale Science and Engineering: The Variational Multiscale Method and Interscale Transfer Operators

*79752*

*R. B. Lehoucq, G. J. Wagner, A. Slepoy, P. B. Bochev, U. Hetmaniuk, G. Scovazzi, M. L. Parks*

## Abstract

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. Our project is interested in preliminary investigations of multiscale mathematics.

We 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.

## Accomplishments

- Continuum-Continuum Multiscale

We explored two approaches for continuum-to-continuum (CtC) multiscale modeling that leverage the flexibility of discontinuous Galerkin methods with the variational multiscale method: local variational multiscale (LVMS) and hybrid continuous/discontinuous Galerkin methods.

The first approach is called the LVMS method in which high-order DG representations are used on each element with a VMS decomposition performed locally on each element. We formulated and implemented this approach within Sandia's high-order DG code called SAGE. The SAGE code is designed to use arbitrary multiscale bases on each element. Since DG readily supports nonconformal elements, neighboring elements need not have the same polynomial order, nor must they possess the same coarse scale representation. In general, distinct multiscale representations can be used on neighboring elements; numerical fluxes are devised that play the role of interscale transfer operators between different multiscale representations on each element. We extensively tested and validated this method for multiscale modeling of wall-bounded turbulent flows. We evaluated the effect of partition selection between coarse and fine scales. We established resolution requirements that are consistent with the near-wall structures in turbulent boundary layers. We used the flexibility inherent in the LVMS approach to perform nonconforming multiscale simulations where multiscale representations are used near the wall that transition to lower fidelity representations outside of the inertial range. Overall, our results indicate that this multiscale approach for turbulence simulation leads to more accurate results with fewer degrees of freedom than traditional subgrid scale models.

The second approach considered is a hybrid continuous-discontinuous Galerkin multiscale formulation that either extends or extracts multiscale representations from more traditional discretizations. Along these lines, we developed multiscale discontinuous Galerkin methods that employ both continuous and discontinuous finite element spaces having much larger dimensions. The continuous space represents the coarse scale approximation, and the difference with the discontinuous space is the fine scale approximation. A standard global discontinuous Galerkin method may be applied to the discontinuous space. The unique feature of the formulation is the use of local, element-wise problems to define the discontinuous field in terms of the degrees of freedom of the continuous field. The local problems employ weakly imposed boundary conditions, and the solutions remain discontinous. However, the local problems are parameterized by the degrees of freedom

of the smaller (in dimension) continuous space. The global problem has the equation size and structure of a continuous Galerkin method but is indeed a discontinuous Galerkin method. The local problems serve to project the solution into a reduced-dimensional subspace that expresses the partial differential equation (PDE) structure of the problem considered. This aspect is related to methods used in wave propagation problems, relying on numerical fluxes inspired by local Riemann solutions, but here the local problems are solved numerically using the local basis functions. Effectively, the local problems give rise to interscale transfer operators and can be interpreted as providing constitutive relations that express fine scales in terms of the coarse scales.

- Atomistic-Continuum Multiscale

In order to describe a body that deforms smoothly and not so smoothly in distinct regions, numerous atomistic-to-continuum (AtC) coupling methods have been proposed. Unfortunately, a mathematical analysis of the effect of the interface used for AtC coupling is only available in the one-dimensional setting. Hence, our accomplishments are preliminary and include reading the available literature.

Our preliminary work included recasting the AtC coupling method found in the literature within the VMS framework. This demonstrated that the VMS framework could represent AtC approaches. Work is under way to determine whether VMS suggests alternate coupling approaches that lend themselves to amenable analysis.

We also explored kinetic Monte Carlo (KMC) techniques for AtC coupling. Coupling KMC to a continuum process extends the capabilities of KMC to significantly larger system sizes. Mathematical identification of the dividing line between the continuum and discrete behavior remains an open scientific problem of great interest.

## Refereed

S.S. Collis and S. Ramakrishnan, “The Local Variational Multiscale Method,” presented and in *Proceedings of the Conference on Computational Fluid and Solid Mechanics*, Cambridge, MA, June, 2005, vol. 3, pp. 623–627.

P. Bochev, T. Hughes, and G. Scovazzi, “Variational Multiscale Framework for DG,” Sandia Report, SAND2005-3334A, Albuquerque, NM, 2005.

S. Ramakrishnan and S.S. Collis, “The Local Variational Multi-Scale Method for Turbulence Simulation,” Ph.D. Dissertation, Rice University, University Press, 2005, SAND2005-2733.

T. Hughes, G. Scovazzi, P. Bochev, and A. Buffa, “A Multiscale DG Method with the Computational Structure of a CG Method,” submitted to *Computer Methods in Applied Mechanics and Engineering*.

S. Ramakrishnan and S. Scott Collis, “Partition Selection in Multi-Scale Turbulence Modeling,” submitted to *Physics of Fluids*.

# Microprocessor Extensions to Accelerate Scientific Applications

*79753*

*K. D. Underwood, K. S. Hemmert*

## Abstract

Recent analysis of Department of Energy (DOE) applications reveals a potential for benefit from microprocessor innovations. This research 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 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 proposal leverages an earlier collaboration with the University of Notre Dame. Data from this collaboration revealed the potential for new microarchitecture features to accelerate DOE applications.

This year, this project revealed further opportunities for acceleration in the types of floating-point operations performed by Sandia applications. As suspected, our research also indicated that traditional benchmarks exhibit some of the properties seen in Sandia applications but do not exhibit opportunities to the extent that Sandia applications do. Our research indicated that integer operations are significantly more important than initially thought, and so we began to investigate how to accelerate the integer part as well. We also created initial ideas for floating-point architectures.

## Accomplishments

The two primary objectives for this year focused on extracting as much information as possible from as broad of a range of applications as possible to maximize our understanding of the architectural requirements of scientific applications. We succeeded in creating a wealth of meaningful data from six Sandia applications (LAMMPS, CTH, ITS, Xyce, MPSalsa, and ALEGRA) and two national laboratories benchmarks (SPPM and a Trilinos driver). Furthermore, we analyzed both the standard benchmark suite for processor design (SPEC) and a data intensive benchmark suite (DIS). We developed tools to analyze the data and extract meaningful properties about the floating-point data dependency graphs and how architecture might be developed to leverage those properties. The results demonstrated that there are dramatic differences between the SPEC suite and true scientific codes that may account for the poor performance experienced by scientific codes on conventional processors. While these results are promising, there is still much to be done to continue to understand the optimal architecture.

The third objective for this year was to create three candidate architectures. Based on the collected data, a primary architecture candidate emerged as a leader, and we discarded other more traditional approaches. We added two additional points in the architecture space for exploration.

The final objective for this year was to evaluate the candidate architectures in emerging technologies; however, this objective was not met this year. However, we accomplished part of an out-year objective (addressing other bottlenecks introduced by the novel floating-point architecture), thanks to a collaboration initiated with the University of Wisconsin. We also began addressing the performance of the integer part of the microarchitecture.

## Refereed

R.C. Murphy, A.F. Rodrigues, P. Kogge, and K.D. Underwood, “The Implications of Working Set Analysis on Supercomputing Memory Hierarchy Design,” presented and in *Proceedings of the International Conference on Supercomputing*, Cambridge, MA, June 2005, pp.332–340.

# Data Mining on Attributed Relationship Graphs

*79754*

*T. G. Kolda, B. W. Bader, J. P. Kenny*

## Abstract

Sandia is uniquely positioned to address the mathematical and algorithmic challenges of large scale knowledge discovery problems arising in intelligence and threat assessment analysis. The analysts often represent data in attributed relational graphs (ARGs), also known as semantic graphs. This representation uses directed edges to encode different relationships between vertices that represent individuals, events, organizations, etc. The challenge is to discover patterns and latent relationships in graphs that encode terabytes of information.

Our goal is to advance the science of data mining by developing fundamentally new mathematical algorithms for graph analysis. Our focus is on graph algorithms, tensor decompositions, and their interconnections. In order to process massive ARGs on parallel computers, we need to develop techniques to efficiently work with distributed ARGs across nodes. Due to the different edge types and interconnection patterns, this presents novel challenges in comparison to interconnection graphs that arise in scientific applications. We propose to develop large scale tensor decompositions and use them for multidimensional latent semantic analysis. Here, the goal is to accomplish a form of dimensionality reduction onto "concept spaces" to help identify clusters and label nodes. We will also develop subgraph identification techniques for matching analyst specified patterns.

Data mining on ARGs has many government and business applications, most importantly in the domain of national security, but also including web search and other day-to-day applications of informatics. If this project is successful, we will build on Sandia's expertise in high performance computing to develop key mathematical technologies for processing massive data sets in the form of ARGs.

## Accomplishments

An extensive literature survey and review in the areas of data mining for homeland security, web search, graph algorithms, latent semantic analysis, tensor algorithms, etc., produced a database of more than 100 entries, including electronic copies of all articles. All members of the team participated in a reading group.

We developed two test platforms in MATLAB for working with tensors. The first platform is for dense tensors. We quickly discovered that in order to work with realistic data, we needed a second platform for larger scale sparse problems. No datatype for storing sparse tensors existed in MATLAB or elsewhere, so we created the new datatype as well as the algorithms. This is a major accomplishment independent of the application to ARGs. For example, in a few minutes on a laptop, we are able to run our methods on a three-dimensional (3-D) sparse tensor of size 50,000 x 50,000 x 70,000 with over 500,000 nonzeros.

We identified the interconnection network of the World Wide Web as a plausible data set to test our algorithms. Nodes represent web pages (or hosts or domains), and edges correspond to hyperlinks. Most researchers treat this as a directed graph, but it is an ARG if the edges are labeled with the anchor text of the hyperlinks. Furthermore, the nodes can be labeled by the type of page, e.g., department page, user page, project page, etc. The critical feature of the web data is that they are large scale, just as in our target application.

On the web data, we have preliminary clustering results using our newly developed multidimensional latent semantic analysis. We expect these methods will help to improve web search, as well as being extensible to ARGs from homeland security applications.

## Refereed

T.G. Kolda, B.W. Bader, and J.P. Kenny, "Higher-Order Web Link Analysis Using Multilinear Algebra," to be published in *Proceedings of the IEEE International Conference on Data Mining (ICDM05).*

## Other Communications

B.W. Bader and T.G. Kolda, "MATLAB Tensor Classes for Fast Algorithm Prototyping," Sandia Report SAND2004-5187, Albuquerque, NM, 2004.

# Multiphysics Coupling for Robust Simulation

*79755*

*R. Hooper, M. M. Hopkins*

## Abstract

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. There exist loose coupling strategies, 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 inter-application 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.

This project is investigating the stability, accuracy, efficiency, and implementation requirements of coupling algorithms to guide application developers in choosing the best coupling algorithms when creating multiphysics applications. We are performing basic research studies on both prototype equation coupling systems (hyperbolic/elliptic/parabolic), as well as real world problems in production application codes. Our findings are driving the development of coupling algorithms that are being made available, along with more traditional coupling approaches, to multiphysics environments via extensions to the NOX nonlinear solver library. They are also being demonstrated in the SIERRA finite element framework's solution control mechanism.

## Accomplishments

We accomplished the following tasks during FY 2005:

(1) We identified a set of prototype applications for basic testing research and for applications. In particular, for the basic algorithmic studies, we identified the HMX cook-off problem used in Sandia's Calore code coupled to a Burgers' equation. We are also using the Brusselator equations.

(2) We identified several real world coupling systems important to Sandia's stockpile stewardship mission and have extensive studies on one of the more challenging ones using the production code Aria within the SIERRA multiphysics framework. This simulation compared loose coupling and matrix-free Newton-Krylov coupling for a microelectromechanical system (MEMS) switch. This involved coupling electrical, thermal, and solid mechanics equations. We made progress in coupling Premo and Calore to solve coupled compressible flow and heat transfer.

(3) In support of applications, we interfaced the NOX nonlinear solver library to Calore and Aria (other supporting codes already have a NOX interface).

(4) We developed a working prototype multiphysics environment to guide and test development of algorithms and software enabling various coupling strategies. We created the prototype environment and the software supporting new algorithms as extensions to the NOX nonlinear solver library, making them readily available as part of the Trilinos 5.0 release.

(5) We crafted several representative coupled problems within the prototype environment and performed several studies.

## Other Communications

R. Hooper, M.M. Hopkins, and R.P. Pawlowski, "Enabling Newton-Based Coupling Within a Multi-Physics Environment Using NOX – An Object-Oriented Nonlinear Solver Library," presented and in *Proceedings of Coupled Problems 2005*, Santorini Island, Greece, May 2005, CD-ROM.

M.M. Hopkins, R. Hooper, R.P. Pawlowski, and S. Gianoulakis, "Coupling Strategies for an Electro-Thermo-Mechanical Actuator," presented and in *Proceedings of Coupled Problems 2005*, Santorini Island, Greece, May 2005, CD-ROM.

# Simulation of Neutron Radiation Damage in Silicon Semiconductor Devices

*79756*

*G. L. Hennigan, E. R. Keiter, H. P. Hjalmarson, P. T. Lin, J. P. Castro, R. P. Pawlowski, J. N. Shadid*

## Abstract

Semiconductor device modeling plays a critical role in the success of the Qualification Alternatives to the Sandia Pulsed Reactor (QASPR) effort. 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 faces several critical hurdles. The exponential gradients of carrier flux and source terms in semiconductor physics require substantially more robust stabilization than is found in flow problems. Also, extreme stiffness due to the wide range of timescales, from nanoseconds for the carriers (electrons and holes) to possibly hours for some 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.

Stabilized Galerkin has been applied with some success to this problem; however, techniques such as mixed element methods and flux corrected transport (FCT) have the possibility of greater efficiency and robustness. Multilevel preconditioning will be necessary for massively parallel scalability. This will be a critical area of our research through application of algebraic multigrid and physics based preconditioners. Finally, development of stiff time integration algorithms, including techniques such as operator splitting, is necessary.

## Accomplishments

We made substantial progress in the Charon device simulator's ability to model semiconductor devices. We demonstrated the ability to simulate stockpile diodes and bipolar transistors with problem sizes approaching 1 million unknowns. Advancements in the semiconductor physics capability, stabilized discretization, and code infrastructure all led to a substantially more capable tool.

The semiconductor physics package has seen substantial expansion in capability. Multiple electrodes with two-dimensional (2-D) doping profiles allow realistic device problems to be easily generated by the user. We can use either experimental depth profiles or 2-D scatter data to define the doping. The automatic mesh refinement capability is now in regular use, allowing an order of magnitude reduction in user effort for mesh generation. This capability generally produces more robust meshes with fewer total elements. We also added multilevel Shockley-Reed-Hall recombination, allowing good agreement with experimental data for a neutron irradiated stockpile diode.

We improved the stabilized Galerkin finite element discretization, allowing greater robustness for semiconductor problems. We now use a tensor based streamline-upwind/Petrov-Galerkin (SUPG) stabilization as well as source term lumping. Convergence studies show second order convergence with substantial improvement for coarse meshes.

We made many new infrastructural changes to add new capability to the tool. We implemented a variable provider layer that allows rapid extension of input data and species handling. The formation now supports colored finite difference and automatic differentiation, including forward and reverse modes, to allow easy generation of sensitivity analysis and problem optimization. We used Chemkin to handle multiple reacting species and will utilize it to support the 30+ species and associated reactions for the candidate neutron defect physics.

# Data Pipelining for Heterogeneous Data Fusion

*79757*

*G. A. Gray, D. M. Gay, K. L. Sale, K. B. Vanderveen*

## Abstract

The development of bioagent detection systems is increasingly important in homeland security applications. Such efforts 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. To meet this need, we propose the development and use of a data fusion technique called pipelining. Pipelining is a computational tool that manages the exchange of information among various data sources and applications. One of its advantages is that it allows data to remain in heterogeneous databases and does not require disparate data types to be converted into one large database. This characteristic prompted biotechnology companies to develop pipelining methods for high throughput data analysis and led to increased success in solving drug discovery and protein identification problems.

Our project focuses on using pipelining to automate the process of data fusion and to extend the use of ensemble classifiers to disparate data sources. We are working to develop an algorithmic framework that will include separate base classifiers for each data type. We are also developing a fusion method for combining the individual classifications and acting as a global discriminator. Due to the approach requiring only the base classification results and not the specific underlying data, pipelining will be accomplished. One of the major challenges of developing such an algorithm is that it currently has no underlying convergence theory. To manage this risk, we are using optimization to introduce mathematical rigor and leveraging Sandia's expertise in algorithmic theory to obtain a provably convergent method. We will accomplish the development and testing of our pipelining software in collaboration with systems research experts, who will provide sample data and applications.

## Accomplishments

Research into current data classification and fusion techniques revealed the existence of an open source public health surveillance software package called RODS (Real-time Outbreak and Disease Surveillance), which is used by Sandians working on Department of Homeland Security (DHS) funded programs. We consulted with Sandia systems experts working in these programs in the design of our data pipelining algorithm. Our algorithm can be described as follows: Disparate data sets, either real or simulated, are input to the appropriate base classifiers. (Some of these classifiers already exist in the RODS, while others will be added.) Then, the resulting base classifications are reported to the fusion algorithm where a global classification is determined and reported.

We identified the following initial data types: over the counter drug sales, school absence records, and hospital emergency room records. We will use both real data and simulated data. RODS currently includes base classifiers for our initial data types, thus we have access to appropriate base classifiers and algorithms for detecting irregularities.

We implemented the unweighted voting fusion method in the RODS framework. It will be used for comparison and to troubleshoot any software integration problems. Because our goal is the fusion of results from disparate data sources, we are investigating a related method, weighted voting. Current implementations use heuristics to determine the weights that are the key to this algorithm. We instead are taking the novel approach of using optimization to define the weighting scheme. Optimization will provide a mathematically rigorous basis for our fusion technique and a provably convergent method of solution. We are investigating appropriate optimization formulations. Moreover, we structured our unweighted voting algorithm so that the addition of a weighting scheme can be done without significant additional coding.

## Other Communications

D. Brown and G.A. Gray, "Implementation of a Data Fusion Algorithm for RODS, a Real-Time Outbreak and Disease Surveillance System," Sandia Report SAND2005-6007, Livermore, CA, 2005.

# Recognizing Patterns in Massive Datasets

*79758*

*P. J. Williams, S. B. Martin, N. M. Berry*

## Abstract

Data mining applications include those problems where it is imperative to classify an underlying state of nature based on analysis of a relevant set of data. Many problems of this type exist in homeland security, military technology, and molecular biology. For example, homeland security analysts may desire early warning, based on volumes of available but noisy data, of the outbreak of bioterror related disease or lethal cargo shipments. The ability to extract knowledge or determine patterns from seemingly inconclusive data is the essence of data mining. The term support vector machine (SVM) is applied to a powerful algorithmic approach to solving the classification problem.

The SVM training problem is solved by optimizing a generalization measure and the ability of the discriminant function to classify the training set. The practical usefulness of the SVM approach is limited by the performance of existing training algorithms. Using the standard formulation of the SVM training problem, a data set comprised of one million samples requires seven terabytes of memory and is computationally intractable even on massively parallel (MP) computers. This project seeks to develop a scalable parallel decomposition algorithm to train SVMs with computational efficiency increased by orders of magnitude for medium and large SVM problems. The new algorithmic approach will decompose the underlying optimization problem into a series of smaller problems, making it possible to train SVMs on massive data sets using parallel computers. A scalable parallel SVM would benefit homeland security, advanced simulation and computing (ASC) program simulation data analysis, and the Genomes to Life program.

## Accomplishments

The project goals were to implement a serial decomposition based SVM with a novel shrinking strategy and select testbed applications. Initially, we focused our efforts on devising an efficient shrinking strategy for the underlying quadratic programming problem. The goal of shrinking is to identify variables at their bounds early in the iterative process and remove them from the problem formulation to produce a smaller quadratic subproblem that can be solved by standard quadratic programming algorithms. We modified an existing state-of-the-art decomposition method to use indicator functions to identify variables at bounds at the solution. Early in the iterative process, heuristics are needed to maintain a valid working set. We compared the computational expense and prediction accuracy of our approach on datasets in the University of California, Irvine's Machine Learning Repository. In addition, we studied the numerical stability of kernel methods to determine the accuracy of kernel based computations in SVMs. Knowing the conditions under which the SVM is unstable can lead to better termination criteria and improved confidence in the solution. We also considered a geometric reformulation of the standard SVM problem.

Our next step was to develop a pattern recognition toolkit, which includes a serial SVM as well as k-nearest neighbor and neural network algorithms. To implement the SVM, we leveraged two Sandia software packages, EPETRA, a parallel matrix library, and OPT++, a nonlinear optimization toolkit.

Finally, we targeted two homeland defense related applications. First, we classified the file types on erased hard disks. Second, we studied the capability of SVMs to reduce false alarms in perimeter security data simulated by the UMBRA framework. We clustered sensors based on global positioning coordinates and then trained SVMs on the data collected within each cluster to obtain a local view of the event detected. Next, we trained an SVM on the local results to compute a global solution.

## Refereed

S. Martin, "Gilbert's Algorithm for SVMs," to be published in *Proceedings of Fifth IEEE International Conference on Data Mining.*

P. Lamborn and P.J. Williams, "Data Fusion on a Distributed Heterogeneous Sensor Network," submitted to the 2006 SPIE Defense and Security Symposium.

## Other Communications

P.J. Williams, "Optimization and the Classification Problem," presented at the INFORMS Annual Meeting, Denver, CO, October 2004.

# Emergent Distributed Tracking and Identification from Features in Wireless Sensor Networks

*79759*

*T. H. Ko, R. C. Armstrong, N. M. Berry, J. J. Carlson, G. Soh, C. P. Kershaw*

## Abstract

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 for this embedded system. As the demands of securing our nation's resources increase, WSN technology is increasingly called upon 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 involving the integration of image sensors in WSNs and in-network processing to conserve bandwidth. This research is intended 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, such as the event representation, that are shared among nodes; this enables the sensor modes 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.

## Accomplishments

To enable accurate testing of the design, we implemented an image data collection system using a small network of XScale microprocessors and commercial web cams to synchronously capture images from multiple image sensors. We gathered several sequences of images from various sensor emplacement maps both indoors and outdoors at different times of the day.

We designed methods for detecting targets reliably with minimal computation, extracting features of the detected targets. Through a survey of the literature, we determined a base set of features to be useful for higher level reasoning, and we identified a larger set for potential future investigation. We investigated two methods for extracting three-dimensional positions of a target and camera position (including orientation): a closed form solution for restricted camera positions and a novel method for full calibration of camera positions. We discovered features capable of differentiating between rigid bodies (i.e., vehicles) and articulated bodies (i.e., animals and humans), and tested an external body of research on human face detection (i.e., AdaBoost).

To prototype this design, we specified the hardware requirements, identified hardware additions to the ERI (Embedded Reasoning Institute) platform node, and modified the ERI platform node to include Agilent Cyclops and Crossbow Stargate. This resulted in a pending patent on the system design, collaboration with Agilent Laboratories, and consultation to the Grand Challenge LDRD Project 81754, "Microsystems Enabled Virtual Perimeter Security," on the future direction of virtual perimeters.

## Refereed

T.H. Ko and N.M. Berry, "Distributed Calibration and Tracking with Low-Power Image Sensors," to be published in *TAPIA 2005*.

T.H. Ko and N.M. Berry, "Distributed Feature Extraction for Event Identification," presented and in *Proceedings of the Ambient Intelligence: Second European Symposium*, Eindhoven, Netherlands, November 2004, vol. 3295, pp. 136–147.

T.H. Ko and N.M. Berry, "On Scaling Distributed Low-Power Wireless Image Sensors," to be published in *HICSS 2006*.

## Other Communications

T.H. Ko, N.M. Berry, D. Shimokawa, and A. Gu, "DISCERN: Distributed Camera Event Recognition Network," presented at the Bay Area Scientific Computing Day, San Francisco, CA, 2005.

# Fast, High Accuracy Evaluation of Technology Insertion Scenarios via Mixed Integer Programming

*93137*

*J. Watson, W. E. Hart, R. D. Carr, C. R. Lawton, D. R. Strip*

## Abstract

Existing techniques for optimization of technology insertion scenarios, e.g., those based on fault tree analysis, omit the impact of part upgrades on logistics operations. In the past, this omission was justified due to the expense of optimizing logistics operations, including part inventory and resource levels throughout supply and repair chains. Recent Sandia projects have developed optimization models of logistics operations based on mixed-integer programming that represent a first step toward more tractable optimization of logistics operations. The primary goal of this project was to significantly accelerate the run times required for logistics optimization. In technology insertion, a large number of scenarios (>> 100) are evaluated to determine the optimal individual selection, necessitating extremely fast optimization of logistics operations to assess the true cost of each insertion scenario. A goal of this project was to assess the type of information that is required for logistics optimization of technology insertion scenarios. This is the first attempt to perform such assessments, and it is likely that qualitatively new types of information, over and above that fed as input to traditional optimization approaches to technology insertion, will be required.

## Accomplishments

The team achieved three primary accomplishments.

The first accomplishment was a dramatic reduction in the run times required to optimize logistics operations for a complex weapons system given a fixed set of proposed technology insertions, i.e., part upgrades. We considered test data based on the Lockheed Martin Joint Strike Fighter, although similar system data from the Future Combat Systems (or any other logistics operations) could be used. The original mixed integer program (MIP) for logistics optimization required 10 hours to solve on a modern high performance workstation. Via a detailed mathematical analysis of the existing MIP, we constructed a new MIP, which, although larger in terms of the number of variables and constraints, was in practice significantly easier to solve. Using a combination of the new MIP and analysis of solver behavior, we reduced the run times from 10 hours to 2 minutes. This result clearly demonstrates the feasibility of assessing the impact on logistics operations of technology insertion scenarios.

The second accomplishment was the formulation of MIP models that explicitly capture the trade-offs between different candidate technology insertion scenarios. A straightforward approach is to solve the MIP for each scenario independently and then select the best. The benefit of this approach is operational simplicity. The drawback to this approach is the number of possible scenarios, which is combinatorially explosive. An alternative is to simultaneously consider all possibilities, obtaining the best scenario via solution of a single MIP. The benefit of this approach is a significant reduction in the number of sub-MIP solves, while the drawback is the size of the single “simultaneous” MIP, which can be difficult to solve in practice. Practical application and comparison will be required to fully assess the trade-offs between these two alternatives.

The third accomplishment was the identification of a number of new data required as input to technology insertion scenario optimization when logistics impacts are considered. These elements are qualitatively different from those considered in traditional approaches to technology insertion scenario optimization, and a number of unexpected modeling issues were exposed during project execution. These issues are crucial in the deployment of the newly developed algorithmic technology. This knowledge has proved useful to existing Sandia projects.

# Electronics and Photonics

## Active Photonic Crystal Devices for Integrated Photonics and Silicon Photonics

*52548*

*J. G. Fleming, J. R. Wendt, W. W. Chow, P. G. Clem, G. S. Subramania, I. F. El-Kady*

### Abstract

This project uses novel silicon based processing techniques to fabricate state-of-the-art three-dimensional (3-D) metallic photonic crystals and investigates the novel properties of these structured materials. At present, Sandia is the only institution capable of fabricating quality samples of these materials. Thus far, we successfully fabricated eight-layer tungsten crystals, with minimum features down to 0.35 µm. There is little published work on metallic systems that display a finite absorption coefficient. Initially, it was unclear whether such samples would exhibit photonic bandgap effects. However, we successfully demonstrated that these novel materials do, in fact, exhibit photonic bandgap effects, and that we can modify the absorption and emission processes by changing the physical structure of the crystals. Modeling is a critical portion of this work. The imaginary component of the metallic dielectric constant causes most codes to fail. Other models treated the metals as ideal, but the properties we are interested in clearly arise from the finite absorptive and emissive properties of real metals. Also, it is unclear how to account for the metal emission process in traditional codes. We made great strides to develop mathematically robust codes and models that begin to take these components into account.

### Accomplishments

In FY 2005, we concentrated our efforts on understanding the thermal emission properties of metallic photonic crystals because this area is the most scientifically and technologically important. Previously, we found earlier work claiming to demonstrate thermal emission in excess of that predicted by Planck's law to be in error. We investigated this problem and found all subsequent experiments to conform to Planck's law. The experiments performed include filament heating and thermal heating experiments.

We made great progress developing a modeling capability for metallic photonic crystals, and we are currently world leaders in this area. However, work must be done in order to couple the model to real world applications in order to more fully understand thermal emission processes.

We also made good progress in the fabrication of 3-D photonic crystals active in the near infrared and visible regimes. This work used electron beam lithography, liftoff processing, and spin-on polymer planarization. The active areas are small but testable, and the process is relatively rapid. The capabilities we developed led to several related projects.

### Refereed

J.G. Fleming, "A Three-Dimensional Photonic-Crystal Emitter for Thermal Photovoltaic Power Generation," *Applied Physics Letters*, vol. 86, p. 249902, 2005.

### Other Communications

I. El-Kady and W.W. Chow, "Thermal Emission from an Active Metallic Photonic Crystal," presented and in *Proceedings of PECS-VI*, Aghia Pelaghia, Crete, June 2005, CD-ROM.

J.G. Fleming, G. Subramania, and I. El-Kady, "Fabrication and Properties of Novel 3-D Metallic Photonic Crystals," presented and in *Proceedings of PECS-VI*,

Aghia, Crete, June 2005, CD-ROM.

G. Subramania, “Fabrication of Omnidirectional Photonic Band Gap Structures for Photonic Devices in the Near Infrared and Visible Frequencies,” presented and in *Proceedings of PECS-VI*, Aghia, Crete, June 2005, CD-ROM.

# Functionalized Nanoparticles for Sensor Applications

*52551*

*D. R. Wheeler, S. W. Howell, S. M. Dirk, M. G. Blain, R. J. Simonson, K. D. Childs*

## Abstract

We are developing functionalized arrays of nanoparticles crosslinked with short conducting molecules that contain sensing functionalities. These bridging ligands modulate their conductivity based on their interaction with analytes. Once assembled between nanogaps and electrodes, this functionalized nanoparticle/organic ligand composite material will provide nanosized sensors that can be easily interrogated. These nanogap sensors will be engineered so that we can fabricate them into arrays of different sensor elements.

This project consists of a number of different milestones that must be met in order to enable the use of functionalized nanoparticles for sensor applications. The first milestone is the appropriately functionalized nanoparticle. The second milestone is a method to assemble the particles. The third milestone is the generation of a nanogap to contain the nanoparticles.

## Accomplishments

We achieved many breakthroughs this year. We developed a method to use bias to control the thickness of films on surfaces in a very controlled manner. This method allows us to assemble films of monolayers to multilayers with excellent precision. We developed materials that have excellent specificity to the analytes of interest (chemical weapon agents) while rejecting common interferrents. For example, we made films that sense dimethylmethylphosphonate but do not sense toluene (a common component of gasoline).

We made improvements to the robotic assembly of our films that allow us to make sensor elements in a very reproducible fashion. We increased testing of the sensing abilities of our materials. We also began work to integrate our sensor into more complex systems so we can evaluate our technology relative to others in our laboratory.

Two of our papers on nanogaps ranked in the initial top ten percent of papers in *Nanotechnology* based on the number of download requests.

## Refereed

S.M. Dirk, S.W. Howell, S. Zmuda, K. Childs, M. Blain, R.J. Simonson, and D.R. Wheeler, “Novel One-Dimensional Nanogap Created with Standard Optical Lithography and Evaporation Procedures,” *Nanotechnology*, vol. 16, pp. 1983–1985, 2005.

S. Howell, S. Dirk, K. Childs, H. Pang, M. Blain, R.J. Simonson, J. Tour, and D.R. Wheeler, “Mass Fabricated One-Dimensional Silicon Nanogaps for Hybrid Organic/Nanoparticle Arrays,” *Nanotechnology*, vol. 16, pp. 754–758, 2005.

S.M. Dirk, S. Pylypenko, S.W. Howell, J.E. Fulghum, and D.R. Wheeler, “Potential Directed Assembly of Aryl Iodonium Salts onto Silicon 100 Hydride Terminated and Platinum Surfaces,” to be published in *Langmuir*.

# Novel In Situ Mechanical Testers to Enable Integrated Metal Surface Micromachines

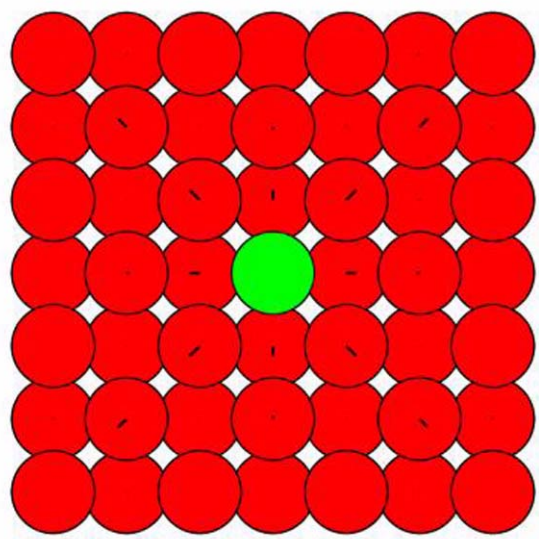
*52552*

*S. J. Hearne, C. W. Dyck, P. G. Kotula, M. P. de Boer, S. M. Foiles, D. M. Follstaedt, T. E. Buchheit*

## Abstract

The ability to integrate metal and semiconductor micro-systems to perform highly complex functions, such as radio frequency microelectromechanical systems (RF-MEMS), will depend on developing free-standing metal structures that offer improved conductivity, reflectivity, and mechanical properties. Three issues have prevented the proliferation of these systems: (1) warpage of active components due to through-thickness stress gradients, (2) limited component lifetimes due to fatigue, and (3) low yield strength. To address these issues, we are focusing on developing and implementing techniques to enable the direct study of the stress and microstructural evolution during electrodeposition and mechanical loading. We are studying the stress during electrodeposition on metal thin films by integrating a multi-beam optical stress sensor into an electrodeposition chamber. By coupling the in situ stress information with ex situ microstructural analysis, we will obtain a scientific understanding of the sources of stress during electrodeposition.

These results are providing a foundation to develop a stress gradient free thin film, directly applicable to the production of freestanding metal structures. We are addressing the issues of fatigue and yield strength by developing novel surface micromachined tensile and bend testers, by interferometry, and by transmission electron microscopy (TEM) analysis. The MEMS tensile tester has a Bosch etched hole to allow for direct viewing of the microstructure in a TEM before, during, and after loading. This approach allows for the quantitative measurements of stress-strain relations while imaging dislocation motion. It also allows for determination of fracture nucleation in samples with well known fatigue/strain histories. This technique facilitates the determination of the limits for classical deformation mechanisms and helps to formulate a new understanding of the mechanical response as the grain sizes are refined to a nanometer scale. Together, these studies will result in a science based infrastructure to enhance the production of integrated metal/ semiconductor systems that will directly impact RF MEMS and LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) technologies at Sandia.



*Displacements of the Ni atoms (magnified 20x) around a substitutional Mn atom calculated using plane-wave pseudopotential calculations using the density functional theory treatment of the quantum mechanics of the electrons.*

## Accomplishments

We made significant progress toward understanding the mechanisms that control stress evolution during electrodeposition of nanostructured materials and Ni alloy films. We also made significant progress toward developing a fundamental understanding of the mechanisms that control fatigue and yield in nanostructured materials.

- Study of stress evolution during electroplating of Au and Cu.

For our study of stress evolution during electrodeposition of nanostructured materials, we focused our efforts on a Ni alloy in order to understand the fundamental sources of stress active during electrodeposition. To this end, our work emphasized the stress evolution during electrodeposition of NiMn from a sulfamate based bath, the materials system currently in use in Sandia's LIGA processes. NiMn stress evolution

with film thickness exhibited an initial high transitional stress region followed by a region of steady state stress with a magnitude that depended on deposition rate. This is similar to the previously reported stress evolution in electrodeposited Ni found in the literature. The incorporation of increasing amounts of Mn resulted in a linear increase in the steady state stress. Microstrain measured by x-ray diffraction (XRD) correlated with the Mn content and the deposition current density. However, we observed no significant changes in the texture or grain size. These results suggest that multiple mechanisms such as twinning, point defect generation, and/or grain boundary coalescence are active in this system. To improve our understanding of these mechanisms, we computed the properties of Mn substitutional impurities in bulk Ni based on first principles density functional calculations. To perform the calculations, we used the Vienna Ab-initio Simulation Package. These are plane-wave pseudopotential calculations using the density functional theory treatment of the quantum mechanics of the electrons.

To address the variation in bond lengths (microstrain) introduced by a Mn substitutional atom, we considered a 108 atom cell, where both the cell volume and the atomic positions were optimized. The distances just under 2.52 Å result from the Ni-Mn pairs, as well as pairs of Ni atoms in the first neighbor shell of the Mn. For the average lattice constant of this cell, the nearest neighbor distance should be 2.498 Å. Thus, these results show that there is a distribution of nearest neighbor distances around this value consistent with the XRD measured strain measurements.

- High cycle fatigue lifetime, and fabricate, test, and analyze nanograined samples.

We directed several efforts toward fabricating the generic samples. Each time, we encountered different issues and did not successfully make samples. We decided to focus on measuring and modeling the SUMMiT V™ (Sandia Ultra-planar, Multi-level MEMS Technology 5) based samples.

## Refereed

S.J. Hearne, J.A. Floro, M.A. Rodriguez, R.T. Tissot, C.S. Frazer, L. Brewer, P. Hlava, and S. Foiles, “Stress Creation During Ni-Mn Alloy Electrodeposition,” submitted to *Journal of Applied Physics.*

S.J. Hearne and J.A. Floro, “Mechanisms Inducing Compressive Stress During Electrodeposition of Ni,” *Journal of Applied Physics*, vol. 97, p. 014901-1, 2005.

S.J. Hearne, S.C. Seel, J.A. Floro, C.W. Dyck, W. Fan, and S.R.J. Brueck, “Quantitative Determination of Tensile Stress Creation During Island Coalescence Using Selective-Area Growth,” *Journal of Applied Physics*, 97, p. 083530-1, 2005.

# An Integrated, Stacked System on a Chip

*52554*

*R. Chanchani, D. W. Peterson, D. Bethke, T. A. Fischer, D. B. Webb, M. E. Warren, D. Galdony*


## Abstract

In this project, we developed a unique concept to integrate different device technologies by stacking wafers in three dimensions. The vision is to build a final product that will be a fully integrated system on a chip including passive and active devices made of different technologies. The uniqueness of this concept, for which a patent application has been submitted, is in:

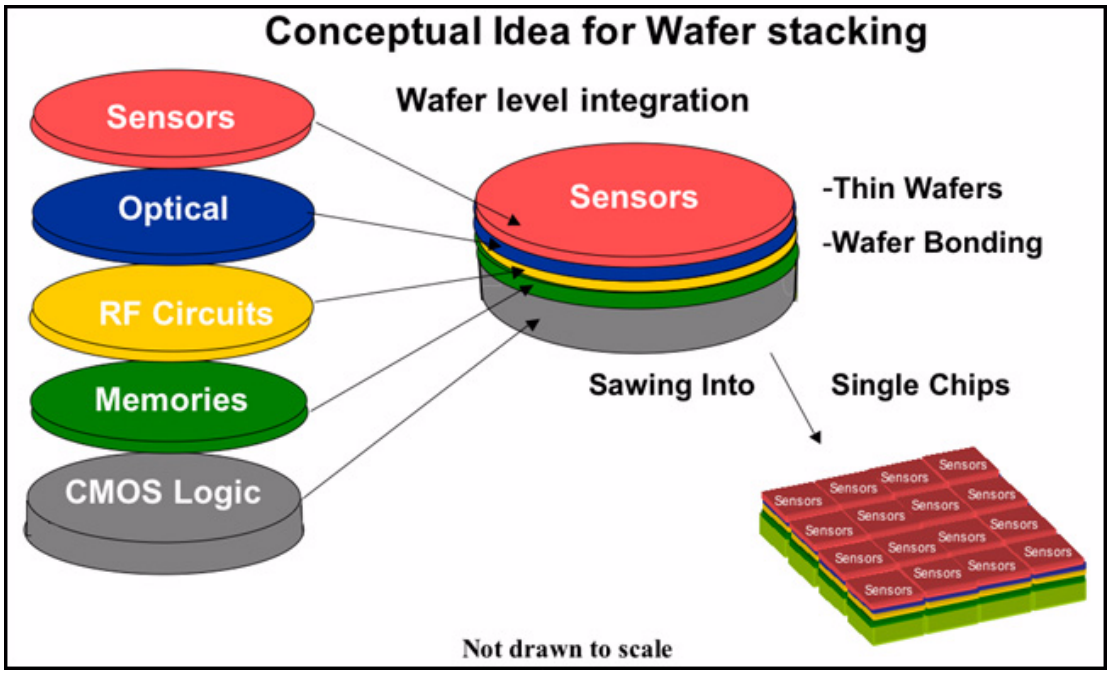
(1) The use of benzocyclobutene (BCB) as a "glue" layer between wafers. This will allow us to incorporate interconnect structure and embedded passives within the "glue" layer.

(2) The use of through-hole vias in silicon with a lower aspect ratio obtained by thinning the top wafer to 20–125 µm thickness.

We developed technology to stack wafers with electrical interconnection between the wafers. We demonstrated this with two wafer stacks using 4" diameter wafers and both two and three wafer stacks using 6" diameter wafers. This technology involved developing a wafer thinning process at a vendor facility, wafer bonding with embedded BCB/Cu interconnect structure, via etching, via isolation, via metallization, and fabrication of daisy chain structures with electrical interconnection between the bonded wafers.

A special test vehicle designed to develop processes and to demonstrate the success of our process consisted of four 50 via daisy chain tests with silicon through-vias of different diameters: 750 µm, 300 µm, 150 µm, and 75 µm in the top thinned wafer. The results show that we get 100 percent daisy chain connection with 750 µm, 300 µm and 150 µm diameter through-hole vias. With 75 µm vias, we get partially good daisy chain connection.

This process involves thinning the top wafer to 110 µm thick by grinding and polishing at various vendor facilities. This thickness was the limit of the thinning capability of existing commercial vendor facilities because thinner wafers without removing the grind damage layer could not withstand post-process handling. The thickness of the top wafer determines how small we can make a through-hole silicon via. Future work will involve using thinner wafers and smaller vias.


*An integrated stacked system on a chip.*

## Accomplishments

This year, we completed the development and evaluation of two and three wafer stacks using 4" diameter and 6" diameter silicon wafers. Specific accomplishments are:

(1) We finished the development of modified processes for 4" two wafer stacks with 110 µm thick top wafers. These improved processes resulted in getting crack free stacks and good electrical connectivity with vias smaller than 150 µm, compared to the via size limit of 750 µm diameter. The improved processes developed are:

(a) A technique to fabricate alignment marks on the bottom face of the bottom wafer. This resulted in excellent alignment during the wafer bonding process.

(b) A bonding process by adding silicon rubber and a Kapton sheet over the wafer stack. This resulted in getting crack free bonded wafers.

(c) A via insulation process using 3000 angstroms of silicon dioxide and 3000 angstroms of silicon nitride.

(d) A via metallization process to get thicker metal film, 6 to 7 µm thick, in the via. The development of this process enabled full electrical connectivity in smaller (150 µm) diameter vias in a daisy chain. This is compared to good electrical connectivity in minimum 300 µm diameter via daisy chains with thinner metal films.

(2) We developed the processes for 6" wafer stacks. We got similar electrical connectivity results with 150 µm diameter vias as for 4" wafers. With 6" silicon wafers, we demonstrated both two wafer and three wafer stacks. We bonded the third thinned wafer on the two wafer stack with two layers of BCB/Cu in the glue layer.

(3) We investigated different commercial vendors for thinning wafers to 50 µm. All except one vendor indicated that they could not thin wafers to 50 µm. The one vendor could thin wafer to 50 µm using a facility in France; however, thinned wafers were very fragile and could not withstand the post-process handling. We initiated discussions with National Security Agency (NSA) engineers, who developed a process to thin wafers to 20 µm thick, which could also withstand post-process handling. This NSA facility closed down, and they transferred the equipment and technology to Sandia, although the transfer did not happen in time to use it in this project.

## Other Communications

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# Micro Mass Spectrometer on a Chip

*52555*

*M. G. Blain, J. G. Fleming, K. L. Linker, D. Cruz, D. E. Austin*

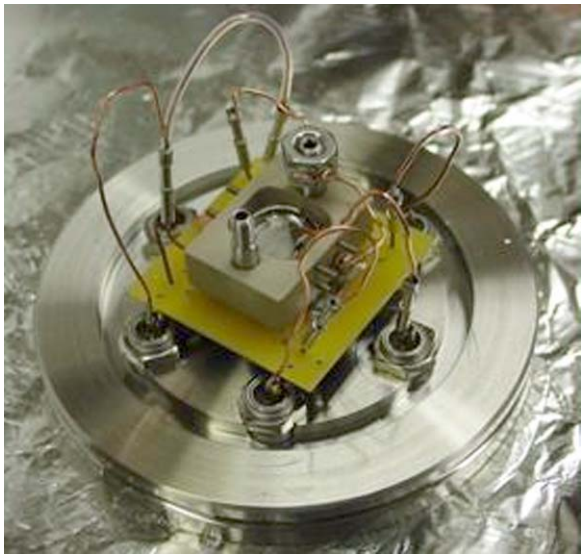
## Abstract

Breakthrough improvements in micro sensors for high consequence applications including chemical and biological warfare agents, medical diagnostics, and explosives detection will be possible only with the development of ultrasensitive and ultraselective microfabricated detectors able to unambiguously identify the compound of interest. A micron scaled mass spectrometer on a chip would set the performance standard for such microdetectors. For example, the combination of a micro gas chromatographic separation column with a micro mass spectrometer would provide a currently unavailable, extremely sophisticated capability for specific and unambiguous molecular identification. A micro mass spectrometer could also serve as a standalone microanalytical system in its own right.

We completed the design, simulation, microfabrication, release, packaging, and testing of prototype micrometer sized cylindrical ion traps (CITs) arrayed for use as micro mass analyzers. We successfully fabricated ion traps consisting of four different trap sizes ($r_0$ = 1, 2, 5, and 10 µm) and three array sizes (from 102 to 106 traps/array) using molded tungsten microelectromechanical systems (MEMS) process technology. We designed 44 different devices across all trap radius and array sizes for functional trap operation, radio frequency (RF) device characterization, and device defect tolerance.

## Accomplishments

We completed fabrication of redesigned CIT mass analyzers to maximize the number of ion traps and to reoptimize the trap size and form factor, in light of ion source and detector requirements. Additionally, we demonstrated functional operation of a massively parallel array of 104 CITs as a mass analyzer that is micrometers in size and built on the surface of ~2 $cm^2$ silicon substrate. Simulation of the as-fabricated trap geometries elucidated the physics of ionizing, trapping, and detection ions in traps of this size. We also understand the implications for mass spectrometry at the limit of one ion/trap in the context of operational pressure and the ionization, trapping, and ejection sequences.



*Micro-ion trap technology enables development of a mass spectrometer on a chip that offers specificity and portability desperately needed by the chemical agent detection community.*

We defined and optimized an operational protocol for ion creation, trapping, and detection with traps of this size. We also designed and implemented an RF package for testing of the ion trap chips. We measured the electrical characteristics of the ion trap arrays, including their resistive, capacitive, and inductive and field emission properties; and equivalent circuits were derived. RF characterization of the traps revealed limitations in device operation due to large trap ring electrode capacitances to ground, resulting from large electrode MEMS anchor areas and from the use of relatively low resistivity silicon substrates. We performed ion trapping experiments at Purdue University's Aston Lab with micro-CIT arrays that were packaged and mounted in an RF circuit board. We were able to attain favorable trapping conditions with an adequate pseudopotential well (trap) depth. We estimated an in situ ionization rate of twice the ion loss rate from measurements and validated simulation results. We obtained evidence suggesting the trapping of toluene ions in operational testing of $r_0$ = 5µm traps; however, performance was severely degraded and could not be

optimized due to limitations. We identified and initiated solutions to these problems. Namely, we achieved an orders-of-magnitude reduction of substrate RF capacitive coupling losses by the use of high resistivity silicon and by the reduction of trap RF electrode anchor area through removal of silicon substrate in the trapping region. The use of high resistivity silicon provides 2 to 3 orders of increased resistivity and provides a commensurate reduction in the capacitive reactance from trap ring electrode to ground. Additionally, the removal of substrate silicon, by putting a through-hole in the Si, should greatly reduce capacitive RF losses. It will also provide optical, ion, and electron access to, and egress from, the trapping region while reducing charge build-up on nearby insulating surfaces.

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# Quantum Coherence in Semiconductor Nanostructures for Improved Lasers and Detectors

*52556*

*J. G. Cederberg, S. K. Lyo, N. A. Modine, W. W. Chow, S. R. Kurtz, R. M. Biefeld, M. C. Phillips*

## Abstract

Advances in atomic physics led to demonstration of several quantum coherence phenomena. These experiments brought worldwide attention, but these phenomena have not been observed in atomic, solid or semiconductor systems. Quantum mechanical coherence and interference can potentially be used in a variety of applications, including lasers, spectroscopy, and optical storage.

The goal of the project is to investigate quantum coherence (QC) effects in semiconductor quantum dots. Technologically this could result in the demonstration of an all-optical delay or an optical qubit in semiconductor self-assembled quantum dots (SAQDs). SAQDs may be better than quantum wells in applications requiring operation at high temperatures. An important consideration is the dephasing lifetime, the time constant for phase retention. In SAQDs, it was measured to be 250 femtoseconds at room temperature. This is in contrast to dephasing lifetimes of 30 to 70 femtoseconds in quantum wells. This lifetime increases significantly at low temperatures for SAQDs to 600 to 800 picoseconds at liquid helium temperatures. Our efforts concentrated on (1) developing and applying a theory for studying semiconductor optical coherence effects, (2) developing and applying a theory for understanding collision effects in SAQD dephasing, (3) preparing SAQDs with a low inhomogeneous broadening, and (4) identifying optimal conditions for experimental observation and the types of experiments needed.

## Accomplishments

Our analytical studies focused on comparing the QD excitation and dephasing behaviors, which we expected to have significant influence in our planned experiments. We developed a quantum-kinetic theory, including memory effects, for this purpose. We found the behaviors to be distinctly different between deep (InAs) and shallow (InGaAs) dots. Our investigation is continuing to determine if deep dot geometry is advantageous to the observation of quantum coherences. We started collaborations with the experimental groups of Diana Huffaker at the University of New Mexico and Connie Chang-Hasnain at the University of California, Berkeley. We are carrying out refinements to the electronic structure calculations jointly with Kaiserslautern University. We are also collaborating with Bremen University to investigate carrier phonon scattering effects. We used the analytical tools developed in the project to evaluate the potential for radio frequency (RF) generation in an airborne platform, jointly with Lockheed Martin. We are presently incorporating the QD theory into a model that can be used for parametric studies of energy transfer between QDs and a polymer host.

Previous research on epitaxial growth of SAQDs enabled demonstrations of InGaAs SAQD heterostructures on the GaAs/AlGaAs platform. This, coupled with the long homogeneous lifetimes observed, suggested that this is a possible system for experimentation. Our desire early in the project was to develop SAQDs that would have optical transitions in the visible or near infrared (emission less than 1 µm) to utilize the optical equipment (sources and detectors) at these wavelengths. Initial growth efforts demonstrated $In_{0.4}Ga_{0.6}As$ SAQDs with room temperature emission at 1075 nm. The problem with these structures is that the inhomogeneous lifetime is as small as 165 femtoseconds (80 meV). The other disadvantage with these structures is that there is no clear excited state observed at room temperature to form the three energy level system needed to observe QC effects. This cooled our interest in $In_{0.4}Ga_{0.6}As$ SAQDs and led us to work to improve the inhomogeneous lifetime of InAs SAQDs. We obtained longer lifetimes of 290 femtoseconds (45 meV) with clear excited state resonances at room temperature. We selected this as the target system for further nonlinear optical investigations.

We investigated several geometries for the optical experiments. We used normal incidence transmission successfully for multiple layers of SAQDs (10 or more layers). This convolves the intrinsic effect of the SAQDs with the effect of assembling many layers. While we investigated this with a single layer, the measured absorbance was not above the noise level. We investigated slab waveguides with angular end facets to

enhance the absorption by increasing the number of passes through a single QD layer. This should have had the desired effect, but any resonances in the signal were still overwhelmed by the background absorbance. Planar waveguides, prepared by polishing crystal edges, proved to be the best geometry for getting large transmission signal from single SAQD layers. We used linear absorbance and photoluminescence to evaluate the energy states of the QDs. The nonlinear measurements enabled by the arrival of an optical parametric amplifier (OPA) to perform the high power pump probe spectroscopy is believed to be the most successful experiment.

In an effort to expand the possible wavelengths where QC effects might be observed, we evaluated the intersubband transitions of SAQDs. We observed two transitions at 7.7 and 9.9 µm. However, the transitions were very broad, suggesting that they were dominated by the inhomogeneous broadening of the sample. Nonlinear spectroscopy may be able to selectively probe only a few of the energy levels available from the SAQD ensemble to enable the separation of the homogeneous contribution to the transitions.

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# Micro Optical Gyroscope via Monolithic Active/Passive Optical Integration

*52566*

*R. D. Kinney, B. Jokiel Jr., F. B. McCormick, J. K. Kim, G. A. Vawter, M. J. Shaw, J. R. Ford, S. L. Shinde, M. Watts, A. Spaltenstein, S. Benson-Lucero, W. J. Zubrzycki, J. J. Hudgens, E. Yepez, J. S. Foresi, T. P. Swiler*

## Abstract

Technological progress in some weapon system related application areas is dependent on the availability of small, rugged, accurate gyroscopes (1–10 degree/hour bias uncertainty). Examples include future W87 Joint Test Assemblies (JTAs) that will require inertial systems with volumes of a few cubic inches to minimize the impact on weapon system dynamics. The gyroscope must withstand reentry environments, yet performance must be sufficient to allow good characterization of vehicle dynamics during reentry.

Optical gyroscopes adopted by industry offer superior ruggedness, reliability, and low cost. However, attempts to miniaturize optical gyroscopes are thwarted by the lack of suitable micro-optical components and monolithic optical integration capability. In the prior years of this project, we developed the photonic elements necessary for a resonant micro-optical gyroscope (RMOG). We individually designed an AlGaAs distributed Bragg reflector (DBR) laser, GaAs phase modulator, and GaAs photodiode detector. The initial challenges we undertook this year were the monolithic integration of these components in a single photonic integrated circuit (PIC), and the reduction of losses in the previously developed silicon nitride planar light circuit (PLC) that implements the optical resonator. We established new goals, including a demonstration of a two chip prototype sensor composed of a PIC and PLC resonator in a laboratory setting. This new goal required a solution for aligning and packaging the PIC with the PLC and the implementation of control and signal processing electronics using laboratory instrumentation. We achieved solutions to these problems.

## Accomplishments

Our accomplishments included the following achievements:

(1) We characterized a 980 nm AlGaAs source/control PIC and a 980 nm PLC.

(2) We contracted with an external vendor, Multiplex, Inc., for the design and fabrication of an InP source/control PIC for low loss operation at a wavelength of 1550 nm.

(3) We designed and fabricated three design iterations of a SiN resonator PLC for low loss operation at 1550 nm wavelength.

(4) We packaged chip pairs consisting of the 1550 nm PIC and PLC to a common header and characterized chip pair performance.

(5) We enhanced our model of the RMOG to incorporate control and signal processing electronics.

(6) We implemented a prototype electronic control system for the RMOG chip pair using laboratory instrumentation to achieve a rate sensor.

Based on our FY 2004 efforts, we decided to focus on developing a 1550 nm wavelength device in InP. This decision was motivated primarily by the need to reduce power loss in the resonator, which has a profound effect on the device performance. We addressed problems of alignment and packaging of the two RMOG chips, the PIC and PLC, as well as the development of an enhanced model, and the implementation of signal processing electronics. We concentrated our internal efforts on further development of a low loss PLC resonator where Sandia's expertise could best be exploited. We contracted for the development of the PIC with Multiplex, Inc., which has substantial experience in InP technology with small linewidth lasers, phase modulators, and photodetectors. This approach proved very productive. We fabricated and characterized several design iterations of the PLC resonator. This could not have been accomplished had the development of the InP PIC been undertaken at Sandia.

We solved significant packaging issues, such as the alignment of the two chips to achieve maximum power transmission from the PIC to the PLC resonator and the bonding and packaging of the two chips to form a single structure. This was a necessary step to achieve a structure suitable for operation as a rate sensor. In addition, we had to address electronic signal processing before the packaged device could be operated as a rate sensor. This required an expansion of the sensor model and the careful selection of appropriate instrumentation for implementing the signal processing system, which we also achieved.

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# Materials Physics and Device Development for Improved Efficiency of GaN HEMT High Power Amplifiers

*52570*

*D. D. Koleske, P. F. Marsh, D. M. Follstaedt, A. A. Allerman, S. R. Kurtz, A. F. Wright, N. A. Missert, C. H. Seager, C. P. Tigges, A. G. Baca, R. J. Shul, P. P. Provencio*

## Abstract

GaN based microwave power amplifiers are identified as critical components to enable Sandia's next generation micro synthetic aperture radar (microSAR) operating at X-band and Ku-band (10–18 GHz). To miniaturize SAR, GaN-based amplifiers are necessary to replace bulky traveling wave tubes. Specifically for microSAR development, highly reliable GaN high electron mobility transistors (HEMTs), which deliver a factor of 10 improvement in power performance compared to GaAs, need to be developed. Despite the great promise of GaN HEMTs, problems associated with nitride materials growth currently limit gain, linearity, power added efficiency, reproducibility, and reliability. These material quality issues are primarily due to heteroepitaxial growth of GaN on lattice mismatched substrates. SiC provides the best lattice match and thermal conductivity, and is currently the substrate of choice for GaN-based microwave amplifiers. Obviously for GaN-based HEMTs to fully realize their tremendous promise, we must solve several challenges related to GaN heteroepitaxy on SiC.

We made a concerted effort to solve these materials issues through in depth research on GaN/AlGaN growth on SiC. Constant feedback between material properties, physical understanding, and device performance will enable rapid progress.

## Accomplishments

We grew approximately 42 GaN HEMT structures on semi-insulating SiC substrates that are suitable for use in radio frequency (RF) electronics, process development, and project device objectives. We used additional wafers to develop material growth and process steps in device fabrication. We discovered how we can vary the metal-organic chemical vapor deposition growth conditions to change the carbon content and dislocation density in the GaN HEMT wafers. Both the carbon content and dislocation density are known to result in electron trapping in GaN HEMTs, and hence are important to RF performance. We studied devices where a thin AlN layer was inserted between the GaN-AlGaN interface and found they result in higher two-dimensional electron gas (2DEG) electron density and higher electron mobility. However, the additional strain introduced by the AlN layer might adversely affect device reliability. We found that changes in the AlGaN cap layer affect the device pinch-off characteristics and change the density of N vacancies and background carbon levels. We found all of these growth conditions to impact device performance. We also studied the critical thickness for fracture and plastic deformation in $Al_xGa_{1-x}N$ alloys ($0.14 < x < 1$) on GaN substrates and found that the strain-relaxation mechanisms in AlGaN alloys place fundamental design limits on the epitaxial heterostructures used in HEMTs for GaN based amplifiers. We developed a model for HEMT device operation that includes transient behavior, effects of passivation, and traps located in the underlying material. Further, we developed a large signal model for the purpose of designing and predicting power amplifier performance. Due to our accomplishments over the duration of this project, Sandia is now capable of achieving greater than 5 W/mm of power density at frequencies up to 10 GHz.

## Refereed

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# Leaky-Mode VCSELs for Photonic Logic Circuits

*52571*

*D. K. Serkland, K. M. Geib, G. M. Peake, W. W. Chow, G. R. Hadley*

## Abstract

Leaky-mode vertical cavity surface emitting lasers (VCSELs) offer new possibilities for the integration of microcavity lasers to create optical microsystems. A leaky-mode VCSEL emits light horizontally, in the plane of the semiconductor wafer, which allows the light to interact with other lasers, modulators, and detectors on the same wafer. The fabrication of leaky-mode VCSELs based on effective index modification was proposed and demonstrated at Sandia recently but is not adequately developed for use in applications. The goal of this project is to advance the design and fabrication of leaky-mode VCSELs to the point where initial applications can be attempted. In the first year, we concentrated on overcoming previous difficulties in the epitaxial growth and fabrication of these advanced VCSELs. In the subsequent years, the focus has shifted to potential applications of leaky-mode VCSELs, such as all optical processing circuits based on gain quenching.

The development of circuits that can process data directly in the optical domain enables encryption and decryption of data on optical fibers at very high rates for secure communications. Such all optical processing circuits will be of interest to the supercomputing and intelligence communities. Additionally, we started preliminary exploration of the use of leaky-mode VCSELs to achieve higher brightness sources, which are relevant to laser radar and remote sensing applications.

## Accomplishments

We continued our efforts to develop new and better methods for the fabrication of leaky mode VCSELs. Additionally, we started work on applications of leaky mode VCSELs to photonic logic circuits based on gain quenching of semiconductor lasers. The final goal was to demonstrate a gain quenched VCSEL, in which an external optical input beam (from another VCSEL) quenches the VCSEL gain and turns off the laser mode. As hoped, we demonstrated gain quenched VCSEL operation.

We continued to refine the fabrication of leaky mode VCSELs. Two device fabrication methods have proven successful. We pursued the first method in collaboration with the University of Wisconsin. This method requires tailoring of the effective index profile by performing a shallow patterned etch (2 to 50 nm deep) just above the cavity layers, followed by epitaxial regrowth of the top distributed Bragg reflector (DBR). We used an InGaP layer to control the etch depth with selective wet etchants and employed a 0 degree miscut substrate in order to achieve the desired vertical regrowth. In this manner, we fabricated large (20 by 20) leaky mode coupled VCSEL arrays. Device heating limited the coherent output.

We also refined our second fabrication method in which we deposited an amorphous dielectric DBR above the etched semiconductor surface. The epitaxial structure was that of a standard 850 nm VCSEL, but with only a 6 period top p-type DBR. We oxidized a quarter wave $Al_{0.98}Ga_{0.02}As$ layer in the first period of the p-type DBR to create a current aperture. We fabricated a two-element coupled VCSEL array by etching two 3 by 4 µm rectangles to a nominal depth of 15 nm. We then deposited an 8 period $SiO_2$/SiN DBR using plasma enhanced chemical vapor deposition (PECVD). We etched the dielectric DBR to access p-type ohmic contacts on top of the semiconductor surface. We demonstrated in-phase and out-of-phase coupling of these two VCSELs, and we recorded output power versus current data that exhibits low threshold currents (1.25 mA) consistent with good quality lasers. Moreover, we recorded emission spectra above and below threshold and compared these with theoretical simulations. Spectral lines from the input optical mode and the quenched optical mode are visible.

We demonstrated a new approach to optical time domain reflectometry (OTDR) that will enable distributed fault monitoring in single mode VCSEL based networks. In situ OTDR uses the transmitter VCSEL already resident in data transceivers as both emitter and resonant cavity photodiode for fault location measurements. Also valuable at longer wavelengths, we demonstrated the concept using an 850 nm oxide confined VCSEL and simple electronics. The dead times and sensitivity obtained are adequate to detect the majority of faults anticipated in local and metropolitan area networks.

In collaboration with the University of Illinois, we produced coupled cavity VCSELs with measured differential quantum efficiencies above 100 percent. In collaboration with Northwestern University, we demonstrated an optoelectronic oscillator using an 850 nm VCSEL for generating low jitter optical pulses.

## Refereed

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# Characterization and Application of Dielectrics with Controlled Leakage

*67022*

*C. W. Dyck, D. J. Dickrell, T. A. Friedmann, M. T. Dugger, J. R. Schwank, J. P. Sullivan, M. R. Shaneyfelt, J. A. Felix*

## Abstract

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 also have lower ohmic losses than micromachined ohmic contacting switches. Presently, these devices are limited by early failures due to buildup of surface and bulk charge during operation.

We propose to solve this problem by engineering novel dielectrics with controlled conductivity to dissipate the 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, and are quantifying the relative contributions of tribocharging and charging from high electric fields. We are considering differences in static and dynamic switch behavior. Analysis techniques include cyclic voltammetry curve measurements, the use of 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.

## Accomplishments

We completed experiments to study the bulk charging characteristics of PECVD films as a function of chemical composition, process methods, and electrical stress. We also completed fabrication and initial characterization of SiON metal-insulator semiconductor capacitors colocated with capacitive switches. We use these structures to correlate bulk charging behavior to capacitive switch performance in an attempt to separate the effects of bulk and surface charging.

Based on our measurements of metal-insulator semiconductor (MIS) capacitors and calculations of switch behavior, PECVD deposited SiON showed the most promise for capacitive switch and other high field applications. This film exhibited the lowest as-processed fixed charge content and lowest electrical stress hysteresis when compared to PECVD deposited SiO and SiN. High quality SiO films with sufficient electrical breakdown were unobtainable, and SiN was eliminated from consideration due to its large hysteresis electrical stress and significant levels of as-processed fixed charge.

We analyzed the chemical composition of PECVD SiON and SiN in order to monitor the consistency of the films, and to correlate the chemical composition of the films to bulk film charging behavior. We also devised extensive cleaning and conditioning steps to improve processing uniformity. The composition of both films stabilized over the course of several months; however, we were unable to correlate these results to charging behavior. We concluded that knowledge of chemical composition is not sufficient to predict charge behavior in PECVD dielectrics, and is thus most likely not correlated to the amount of chemical bonding, such as Si-H, that is known to be responsible for bulk trapping. Fourier transform infrared spectroscopy would probably be a more effective analysis technique.

We performed cyclic voltammetry measurements of the wet and dry processed SiN and SiON samples. The SiN samples had higher as-deposited charge and hysteresis, as expected; however, they were insensitive to the plasma ashing step. On the other hand, the SiON samples were deposited with lower residual charge and had less hysteresis, but were sensitive to the ashing step. We are still investigating the causes of this behavior.

We began measurements of SiON metal-insulator semiconductor capacitors colocated with capacitive switches. Initial results of measurements made in nitrogen and air indicate that substantially greater lifetimes are obtained in nitrogen, underscoring the importance

of a hermetic environment during the operation of these devices. This also suggests that dielectric surface plays a significant role in switching lifetimes. Switches actuated with electric fields in the range of 1 MV/cm failed open circuit, and switches actuated with electric fields in the range of 2 MV/cm and greater failed by sticking down. This was true for both air and nitrogen ambient atmospheres. We also measured the MIS structures, and we are currently correlating these results to the switch behavior.

We submitted some of these test structures for radiation testing; however, equipment problems and malfunctioning switches resulted in ambiguous results.

We fabricated a second set of novel MEMS test structures using aD as the switch dielectric. The breakdown voltages of these films was substantially below 1 MV, resulting in breakdown on every capacitive switch device we tested. We fabricated aD MIS capacitors on silicon substrates and tested them in preparation for irradiation studies, but the results were ambiguous due to excess current leakage. We made the decision not to irradiate these structures.

# Nano-g Accelerometers Using Nanophotonic Motion Detection System

*67023*

*D. W. Carr, L. L. Hunter, G. R. Bogart, U. Krishnamoorthy, T. D. Osborn, R. H. Olsson, A. A. Talin, N. A. Hall*

## Abstract

We continue to make a great deal of progress in the development of a new grating based motion detection system for application in inertial sensing systems. This technology has the potential to detect gravitational changes induced by the motion of the moon around the earth. We successfully fabricated near field coupled gratings that behave consistently with the predictions, and we are making steady progress towards the anticipated minimum force detection below 1 nG $Hz^{-1/2}$.

This device will require features that are much smaller than the wavelength of incident light used for the detection. We use extreme ultra violet lithography (EUVL) and deep ultraviolet lithography for the fabrication of these grating structures. A significant part of this effort involves the development of processes for integrating the EUVL capabilities into microsystems fabrication capabilities.

## Accomplishments

We worked toward the goal of producing an accelerometer system based on our optical transducer designs. After performing a thorough analysis on the transducer devices designed and fabricated in FY 2004, we designed a new set of devices. We completed a design run incorporating the new device designs utilizing the same process previously developed. The devices help us gain more knowledge about the optical properties while also bringing us much closer to having functional accelerometer systems. However, additional process development is needed in order to incorporate better flexural elements, as well as much larger masses, to meet the aggressive demands of this system. For this effort, there are two new processes under way that involve post-processing of wafers.

In parallel with the wafer processing, we are trying to learn more about the fundamental capabilities and limitations of optical sensing of low frequency accelerations. The long lead time of processed wafers enabled us to explore this through the use of conventional components to build a Michelson interferometer with spring mounted mirrors. The macroscale interferometer seismic test-stand (MIST) was extremely helpful in understanding the sources of noise associated with the laser and the electronics. We designed our own electronics board that subtracts the laser intensity noise at the level of the photocurrents; this enables a high gain amplifier circuit for this measurement. This system realized a noise floor below 10 nG $Hz^{-1/2}$ at 1 Hz, and was able to detect signals at the level of tidal accelerations. All of the knowledge gained with MIST will feed into the design of the nanograting based system that is being assembled.

At the same time, we continue to develop the advanced lithography processes. With the decommissioning of the EUVL system, this effort transitioned to nanoimprint lithography, a much more promising technology for nanophotonic device development. We are using a recently acquired system to develop the processes of making imprint masters and to develop pattern transfer from the imprinted structures. We will then use this knowledge to make novel optical and nanomechanical structures that could be useful in future transducer designs.

# Bragg Fiber Development

*67024*

*J. G. Fleming, S. S. Mani, G. R. Hadley, M. C. Phillips, G. S. Subramania*

## Abstract

This project targets the development of a novel approach that allows light and fluids to be confined within the same microvolume. This technology will be capable of more efficient interaction of the light with the fluid and more efficient detection of any emitted light. The devices created will be able to be arranged in arrays on chip. One promising application of this technology will be the development of much more sensitive, miniaturized biological detectors. The technology is based on the concept of Bragg fibers. Whereas fiber optic cables trap light in a high index of refraction medium by total internal reflection, Bragg fibers trap light in a low index of refraction medium surrounded by distributed Bragg reflectors. This is significant, as biological fluids generally have a low index of refraction. Although proposed over 20 years ago, Bragg fibers have only recently been fabricated. Sandia was the first to demonstrate a range of structures, mirror stacks and waveguides, built using micromachining to produce the Bragg fiber cavities. This previous work used air as the low index medium. In this project, we will use fluids as the low index medium inside the Bragg fiber. As a demonstration vehicle, this project will focus on demonstrating a Bragg fiber micro-optic/microfluidic system for optimizing the detection of fluorescent "tags" that are selectively attached to specific biological compounds. This technology also has the potential for spectroscopy and light induced chemistry applications. In the course of this work, we found that these structures may enable the creation of very high Q (quality factor) cavities. This is potentially very interesting for various sensing and integrated optics applications.

## Accomplishments

In FY 2004, we developed and widely published the theory behind Bragg fibers and Bragg onions. In FY 2005, we worked to characterize fabricated structures. We concentrated on visible emissions of interest to biological studies. We conducted detailed measurements of mode shape and propagation loss in the visible spectrum. We also developed a novel and more rapid characterization technique based on the output of a microscope color video display. We successfully demonstrated the ability to obtain accurate data on mode shape and intensity using this approach. The advantage of this approach is that it is much simpler than laser based testing and lends itself more readily to the testing of liquids. In initial testing with water, we ran into difficulties with air bubbles blocking the light path. However, in the course of further modeling and experimental work, we determined that it is possible to have reasonable transmission through the Bragg fiber even if the fiber top is partially open. We hope to use this result, and larger fibers, to address the difficulties we have in testing liquids in the fibers.

## Other Communications

J.G. Fleming, M.C. Phillips, D.L. Luck, and G.R. Hadley, "Guiding of Visible Light Using a Lithographically Patterned Bragg Fiber," presented and in *Proceedings of PECS-VI*, Aghia, Crete, June 2005, CD-ROM.

# Microwave to Millimeter-Wave Electrodynamic Response and RF Applications of Semiconductor Quantum Nanostructures

*67025*

*M. Lee, R. G. Dunn, J. A. Simmons, J. L. Reno, E. A. Shaner, C. Highstrete*

## Abstract

We continued research into the electrodynamic properties of semiconductor quantum nanostructures in the frequency range 0.1 to 150 GHz. We exploited this knowledge to prototype innovative high frequency devices that have radically superior performance that results explicitly from quantum effects. Specifically, we made significant progress in three areas. First, we finished developing the necessary infrastructure for measuring quantum effects at high frequency with the successful installation of a vector network analyzer and cryogenic probe station and a microwave compatible cryogenic He-3 probe. Second, we significantly advanced the understanding of nonlinear frequency mixing by resonant plasmon excitations in GaAs-AlGaAs semiconductor quantum wells by making a full bandwidth measurement of the intermediate frequency (IF) bandwidth in these structures. The result shows that operating as a nonlinear mixer in response to frequencies near 145 GHz, the plasmon excitations mix with an IF bandwidth of 8 GHz, which is close to the plasmon lifetime as measured by the quality factor of the plasmon resonance. Third, we initiated work on studying the microwave electrodynamic response of semiconductor carbon nanotubes (CNTs). We developed a test-bed geometry compatible both with nanotube directed assembly and with broadband (0 to 100 GHz) phase sensitive electrodynamic measurements. We took the first existing initial measurements of the broadband alternating current conductivity of single walled CNTs.

## Accomplishments

We installed, tested, and calibrated a complete vector network analyzer (VNA) system based on Agilent Technologies' E8364B and 4155C analyzers. This VNA is capable of measuring complex S-parameter linear response and some frequency conversion characteristics from 0.010 to 50 GHz. It is fully integrated with a cryogenic probe station capable of holding device under test (DUT) temperatures between 4 and 300 K. The system has a full 50 GHz of test bandwidth. A National Institute of Standards and Technology traceable set of calibration standards is used to maintain precision and accuracy to better than 1 percent absolute.

We completed a cryogenic probe for high frequency measurements in an existing He-3 refrigerator and high field magnet system. The refrigerator is capable of generating temperatures as low as 0.3 K and magnetic fields up to 12 Teslas. These conditions are necessary to produce quantum response in quantum point contacts and quantum dots. This probe has two semi-rigid coaxial cables with K-connectors capable of carrying frequencies up to 40 GHz to and from a cold DUT.

We fabricated a quantum point contact (QPC) nanostructure on a high mobility two-dimensional electron gas GaAs-AlGaAs heterostructure. At a temperature of 0.3 K, this device showed a series of well defined source-drain conductance steps as a function of the point contact gate bias, each step at an integer multiple of the fundamental conductance quantum. This indicates a very high quality QPC, where the number of electron wavefunction modes crossing the point contact can be precisely controlled via the gate bias.

The main improvement to the initial generation of quantum well (QW) plasmon mixers was to replace the wire-bond source and drain contacts with integrated coplanar waveguide (CPW) electrodes. The great advantage of CPW contacts is that they are compatible with coupling out signals up to a very high frequency, > 100 GHz in the case of our design, without parasitic inductive choke caused by wire bonds that limit device frequency performance to < 2 GHz. Using the CPW integrated QW plasmon mixer, we substantially improved our previous technique and succeeded in measuring the full intrinsic intermediate frequency (IF) bandwidth of the mixer when biased on a plasmon resonance. We found the -3 dB IF bandwidth on resonance to be about 8.5 GHz when mixing down a 145 GHz local oscillator. This IF bandwidth is essentially the data rate bandwidth of the mixer. That is, if we used this mixer as a communications receiver, it could support a data rate of about 8 Gbits/s. An 8.5 GHz IF bandwidth is, relatively speaking, very fast. Commonly

used Schottky mixers typically have 5 to 10 THz of IF bandwidth. Interestingly, the IF bandwidth is roughly the same as the frequency half-width of the plasmon resonance, suggesting that the IF bandwidth is physically limited by the plasmon lifetime in the QW mixer.

We successfully designed and fabricated a broadband high frequency device platform for measuring the microwave electrodynamic response of directly assembled CNTs. Preliminary measurements show that we can measure the reflection and transmission from a small number of individual CNTs across a bandwidth of 50 GHz.

## Refereed

M.Lee, "Phase Diagram of Coulomb Interactions Across the Metal-Insulator Transition in Si:B," *Physical Review Letters*, vol. 93, p. 256401, 2004.

M. Lee, M.C. Wanke, and J.L. Reno, "Millimeter Wave Mixing Using Plasmon and Bolometric Response in a Double-Quantum-Well Field-Effect Transistor," *Applied Physics Letters*, vol. 86, p. 033501, 2005.

## Other Communications

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, "Single-Quantum-Well Grating-Gated Terahertz Plasmon Detectors," to be published in *Applied Physics Letters.*

# Development of GaN 20-100 Watt Ku-Band Power Amplifiers for MicroSAR

*67026*

*A. G. Baca, M. E. Overberg, J. R. Wendt, M. Armendariz, R. J. Shul, P. F. Marsh, C. E. Sandoval, S. L. Shinde*

## Abstract

GaN solid state power amplifiers (SSPAs) are promising candidates to replace traveling wave tubes (TWTs) currently used in synthetic aperture radars (SARs). A GaN SSPA can result in a 40x size reduction and a 5–7 lb. weight reduction of primary components (plus secondary gains from placement), and a 40x cost reduction. In addition, GaN SSPAs can offer the combination of high power levels and fast dynamic switching for radar fuze applications. For SAR applications, TWTs provide power levels from 5 to 100s of watts at Ku-band. GaAs solid state solutions are available below the 5 W power level at the bandwidths required for radars. For S-band (2–4 GHz), GaAs devices can deliver up to 10 W in radar applications. However, due to the lower power density of GaAs, large gate periphery devices with high gate capacitance are required. The high gate capacitance of the GaAs devices will result in slower switching than desired between the transmit and receive modes in a radar. Due to favorable semiconductor properties, such as high breakdown field, high charge density, and good thermal conductivity, GaN demonstrated 10x higher power density results compared to other semiconductors. GaN SSPA technology provides approximately 10 times higher power density than GaAs. One can exploit these advantages to realize higher total power in SSPAs, or faster switching, lower gate capacitance SSPAs at comparable powers. This will enable greater solid state power in SARs and faster dynamic switching in other radars.

We will advance the state of the art in high frequency total power, as well as exploit the unique advantages of GaN in power amplification with dynamic switching. We will address difficult problems associated with achieving these goals, namely, thermal management, device characterization, device stabilization, power combining, and packaging.

## Accomplishments

We built and characterized GaN high electron mobility transistors (HEMTs) for a 20 W, 3 GHz SSPA with direct current, radio frequency, and load pull. We extracted a large signal model for GaN HEMTs with a 0.6 µm gate length and a 1.2 mm gate periphery. We extrapolated the characterization data and models for a larger 6.0 mm periphery GaN HEMT and designed matching circuits for a 20 W, 3GHz SSPA. We fabricated and tested transistors. We then designed the SSPAs using the advanced design system software for high frequency design. Next, we built and characterized matching and bias circuits. Finally, we assembled and tested the amplifier. The amplifiers yielded 43.2 dBm (21 W) of continuous wave power output at 2.7 GHz. We used the same size transistor to design a 20 W drain switch. This design is still in progress and needs to be optimized and finalized for realization of a pulsed 20 W SSPA.

## Other Communications

A.G. Baca, Y.M. Kim, P.F. Marsh, M.G. Armendariz, C.E. Sandoval, A.A. Allerman, S.R. Kurtz, M.E. Overberg, J.R. Wendt, and C.A. Sanchez, “Pulse Testing of GaN/AlGaN HEMTs,” presented and in *Proceedings of the State-of-the-Art Program on Compound Semiconductors XLI and Nitride and Wide Bandgap Semiconductors for Sensors, Photonics, and Electronics V*, Honolulu, HI, October 2004, PV2004-06, pp. 435–440.

# Evanescent Wave Planar Photonic Biosensor

*67027*

*D. W. Branch, M. J. Shaw, G. A. Vawter, I. Brener*

## Abstract

The bioterrorism threat to national security has created an increased and immediate need for effective biosensors that rapidly assess the presence and nature of a biological attack. In many cases, the only way to unambiguously differentiate between a hostile attack and a naturally occurring appearance is through the forensic analysis of deoxyribonucleic acid (DNA) sequences present in the suspect agent. While the biotechnology exists today to perform this analysis, current methods rely on polymerase chain reaction (PCR) 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 a unique identifying base sequence, and would be capable of distinguishing near matches that differ by only one base pairing, i.e., single nucleotide polymorphisms (SNPs).

We propose 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 will employ guided wave structures enhanced by the addition of ring resonators. Overall sensitivity will be further enhanced by using gold or quantum dot nanoparticles as the attached optical reporter tags to amplify the optical response, conceivably to the point of being able to detect as little as one bound DNA strand.

## Accomplishments

Guided wave devices are a unique technology to interrogate fluid properties and operate as high density immunoassays for the detection of biowarfare agents. Through our work on low loss, high $\Delta$ refractive index, $Si_2N_3/SiO_2$ planar lightwave circuits, we realized microring resonators with quality factors (Qs) greater than 2.4 $x10^5$, fabricated in a standard complementary metal-oxide semiconductor (CMOS) production facility. To that end, we optimized waveguide thickness to minimize loss, allowing us to proceed with fluid sensitivity measurements. We designed and fabricated a Mach-Zehnder interferometer with a microring attached to one arm to measure the induced phase change by the ring resonator. A critical step was controlling the thickness of the guiding region above the rings to maximize sensitivity. We also designed a fluidic microchannel that permits selected delivery of two distinct fluids to the microring resonators. This level of packaging permitted rapid assessment of detection limits while permitting us to reuse the devices. Coupling diode sources to the evanescent device, so that the resonant frequency can be monitored during antigen capture or for fluid detection experiments, is critical to device operation. We also experimented with additional conjugation approaches to improve film stability for antigen capture. We are currently evaluating several chemical attachment methods using organosilane-epoxide based linkers for receptor immobilization. Lastly, we finalized the device and readout procedure such that we can examine the mass sensitivity for a variety of agents on the benchtop.

## Refereed

J. Guo, M.J. Shaw, G.A. Vawter, G.R. Hadley, P. Esherick, and C.T. Sullivan, "High-Q Micro-Ring Resonator for Biochemical Sensors," presented at SPIE Photonics West, San Jose, CA, January 2005.

# Passive Electronically Steerable Array for Miniature Synthetic Aperture Radar, Precision Guidance, and Intelligence/Surveillance/Reconnaissance

*67028*

*K. C. Branch, B. H. Strassner II, T. A. Plut, C. D. Nordquist, C. W. Dyck, G. M. Kraus*

## Abstract

The overall project goals are to develop an antenna system suitable for miniature synthetic aperture radar (miniSAR) systems and to facilitate radar use for precision guidance, tactical unmanned aerial vehicles, and other intelligence/surveillance/reconnaissance applications. The proposed antenna system is a passive array constructed without expensive and complex transmit/receive modules. Instead, a microelectromechanical system (MEMS) phase shifter is inserted into the antenna feed path, creating a compact, low loss, lightweight, passive electronically steerable array (PESA). We are making progress both in technology development and integration. Our work continues to evolve as we test proof-of-concept prototypes and discover issues that will require further research.

At the device level, we improved the MEMS ohmic switches to produce yields of ~97 percent. Our greatest challenge is developing phase shifters with low enough losses that would permit hundreds of them to be integrated in an array system. Simultaneously, we modified the 16 element antenna array to produce sidelobes 35 dB below the main beam. We used these modular wide bandwidth antenna cards to assemble a two-dimensional (2-D) array. Although this assembly does not contain phase shifters, it is an early and promising precursor to the 2-D ESA envisioned by project end.

## Accomplishments

We demonstrated the PESA concept during preliminary tests. The project timeline gave us enough latitude to choose the most appropriate technologies and components. Thus, one of the goals of this second year was to downselect technologies, modify milestones, and choose the final path forward to the 2-D PESA in year three.

We improved the MEMS ohmic switch to the point that a capacitive switch is no longer needed. Switch yields dramatically improved to ~97 percent. We redesigned and tuned the second generation phase shifter fabricated in FY 2004. A third generation 4-bit phase shifter, including a custom glass hermetic package, is currently in the design process. At the antenna level, we modified the 1 x 16 array from FY 2004 so that its Taylor weighted feed structure produced lower sidelobes, 35 dB below the main beam (necessary for many applications, such as SAR). Other antenna developments include the assembly of a 2-D modular card antenna array system.

While we successfully demonstrated the concept with the 1-D PESA, it was limited by the lossy and mismatched interconnect between the phase shifter and antenna and the poor return loss of the phase shifters themselves. Thus, we did not expect (nor did we see) high gain, a narrow beam, or low sidelobes. Since the mismatched amplitude and phase of the phase shifters spread the main beam, beam steering is not as easy to discern. The 45 ° phase increment between each pair of antenna elements should theoretically yield beam steering 7 ° off the main axis. The beam steering observed in the measurements matched this target value. We built this 1-D PESA demonstration with previously developed technologies. Due to the advancements and analysis, the next prototype will have several advantages: (1) one phase shifter per antenna element (thus 16 total phase shifters rather than 8), (2) a custom glass package for the phase shifters, (3) coplanar waveguide interconnects (no wire bonds), and (4) a board for the direct current control lines (rather than external wires). We expect that with these improvements, the antenna array performance will drastically improve and the development of a 2-D ESA is still an attainable goal.

Our activities and accomplishments include:

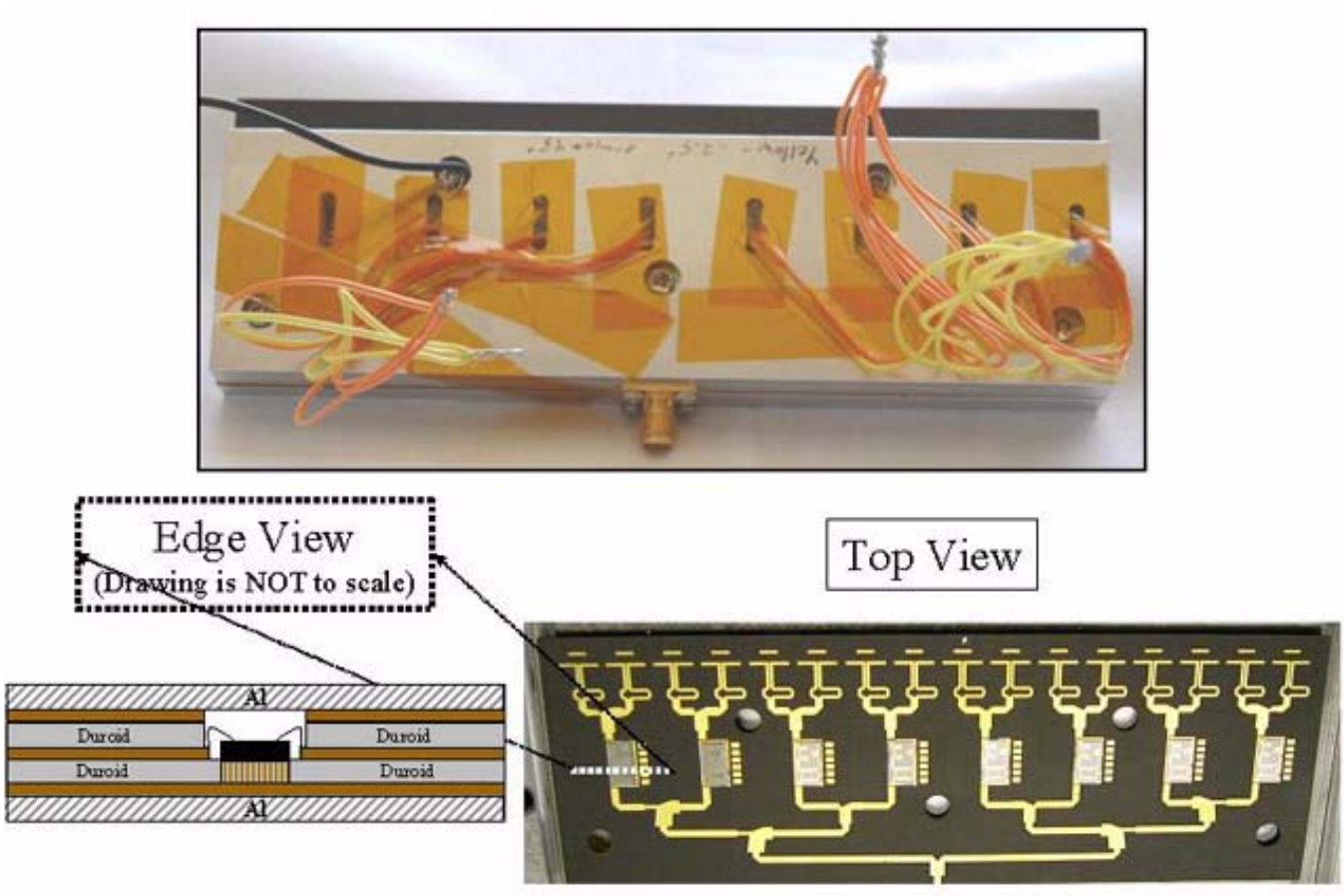
(1) We demonstrated a 4-bit phase shifter.

- Insertion loss average (16.7 GHz) = 1.45 dB (including launches)
- Return loss (16.7 GHz): 9.7 dB
- Switch yield: ~ 97 percent
- Phase shifter yield: 10 of 14

(2) We demonstrated glass-cap packaging of MEMS switch (proof-of-concept for custom packaging technology).

(3) We tested antenna performance of a 1 x 16 array with Taylor weighting.

(4) We tested antenna performance of a 16 x 16 antenna array.




*First PESA prototype.*

(5) We completed phase shifter tuning and redesigned the coupler.

(6) We assembled a 1-D antenna with unpackaged phase shifters.

(7) We tested antenna performance of a 1-D antenna array with unpackaged phase shifters.

(8) We reviewed design of the 4-bit phase shifter.

- The insertion loss of the coupler was too high. Since the coupler is intrinsic to the reflect-line architecture, the decision was made to change the phase shifter architecture.
- We analyzed alternative phase shifter designs.
- We performed comparison between true-delay and true-phase schemes.
- We scheduled and planned prototypes for FY 2006.

## Refereed

C.D. Nordquist, C.W. Dyck, B. Jokiel, T. Lemp, G.M. Kraus, T.A. Plut, W.D. Cowan, C.T. Sullivan, and T. Lemp, "High Performance Packaging of RF MEMS Switches and Circuits," presented and in *Proceedings of the 2005 GOMACTech Conference Digest*, Las Vegas, NV, April 2005, vol. 1, pp. 316–319.

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# Novel Photonic Crystal Cavities and Related Structures

*79760*

*J. G. Fleming, G. S. Subramania, I. F. El-Kady, W. W. Chow*

## Abstract

Photonic crystal microcavities are the photonic analogue of doped semiconductors. Controlled doping changed semiconductors from a scientific novelty to a multibillion dollar industry. In a similar sense, we now have the ability to fabricate high quality one-, two-, and three-dimensional photonic crystals, and one of the next key steps to transitioning to useful devices is the ability to create well controlled cavities and related structures. It is also the case that future photonic devices will not be simple planar semiconductor structures, but will rather develop as complex combinations of emitters, like nanostructured elements such as photonic crystals and novel materials such as metals, that are not traditionally considered in light emitting systems. The experimental development of these devices will be greatly enhanced by coupling to a theory that accurately describes the emission physics and related optical nonlinearities of a photonic crystal structure capable of incorporating novel materials, such as metals. We developed such a microscopic theory. Further refinement and application of this theory are important aspects of this project.

## Accomplishments

We made significant progress on the modeling tasks of this project. In particular:

(1) We drove the analytics of a fully discretized set of Maxwell's equations on a fourth order accurate platform in time domain.

(2) We drove the necessary generalization conditions for inclusions of electrically and magnetically dispersive materials.

(3) We tested the analytical equations for accuracy against problems with known solutions.

(4) We proceeded to generate a numerical code incorporating the necessary computational packages for functioning on a high performance computational platform.

We also fabricated and tested a wide range of samples, including three-dimensional photonic crystals with a range of simple cavities. We found differences between different designs but have yet to compare the results to theory. We fabricated one-dimensional structures to test the modification of Er emission processes; these structures await testing. Recently, cavities based on symmetry, rather than "photonic bandgap" confinement, have been proposed. We fabricated a range of such structures; when tested they do seem to have a complex spectra that we have yet to tie to the proposed symmetry effects. Finally, we investigated novel split ring resonator structures at radio frequencies using commercially available electromagnetic modeling packages. This approach to cavity formation may be attractive to certain applications.

# Integrated NEMS and Optoelectronics for Sensor Applications

*79761*

*D. W. Carr, D. K. Serkland, K. D. Childs, G. R. Bogart, T. O. Townsend*


## Abstract

This project takes on the challenging issues involved in the integration of nanoelectromechanical systems (NEMS)/microelectromechanical systems (MEMS) and optoelectronics to make compact sensor systems, with the primary goal being a very small (~5 $mm^3$) accelerometer with nano-g sensitivity. Besides the clear benefits of size reduction in a single sensor system, another motivation for pursuing this miniaturization is to more easily fabricate sensor arrays. Multiple sensor elements can be used for noise reduction and system robustness in inertial sensing. Arrays of acoustic sensor elements can also be used for source localization and separation, and so in many ways, this research is foundational in nature and will feed into many related programs that are growing from optical based sensing solutions. The focus is on understanding methods of integration, as well as the physical challenges that are involved in closely coupling active optical components, such as lasers and photodiodes, with moving structures that can be used in physical sensors. In this first year, we developed a concept for making a very compact accelerometer system using a diffraction based mechanical sensor coupled to a vertical cavity surface emitting laser (VCSEL) source. We performed a careful analysis of the different ways in which these pieces can be brought together and of the types of sources and detectors that are best suited for this effort. We also developed ways to integrate a proof mass with the optical transducer component and began testing our first devices.


## Accomplishments

We selected a design that integrates a laser, photodiode, and an optical transducer in a single package. We need to work with a design that avoids the effects of laser feedback. We do this by positioning the sensor at an appropriate angle; thus, we can operate in reflective mode without any signal returning to the VCSEL. This system also employs a diffraction approach that enables differential detection in a small form factor. This effort also utilizes existing devices fabricated for a previous effort, adapted to inertial sensing. We chose to work with off-the-shelf photodiode components, as these can be acquired in bare die and in very small form factors.

We developed a method of integrating a large proof mass onto the structure using a solder ball attachment, and this has shown promising results. We took the initial thin film acoustic sensor with a resonant frequency of 91 kHz and reduced this to 2 kHz with the addition of the mass. This reduction in frequency leads to a sensitivity enhancement that is dependent on the square of the resonant frequency. Ideally, we would like to have this below 1 kHz in order to have the sensitivity required for 1 ng acceleration detection.

We also produced a compact VCSEL and photodiode system that can be used for testing and integration with other devices. This includes a VCSEL, a microlens, and a detector combined together in a 14 pin dual in-line package. We will be using this device to investigate the optical performance of recently fabricated grating transducer structures that are optimized for functionality at the 850 nm wavelength of the VCSEL sources.

# Development of Advanced UV Light Emitters and Biological Agent Detection Strategies

*79762*

*M. H. Crawford, A. A. Allerman, K. H. Bogart, A. J. Fischer, R. L. Schmitt*

## Abstract

Biological warfare agent detection with high sensitivity and low false alarm rate is a critical capability for national security. The ideal bioagent detector would be compact, robust, low maintenance and would supply 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 (1) the lack of compact, robust, efficient ultraviolet (UV) light sources at optimal wavelengths; and (2) the inability to effectively discriminate against background signatures in a compact system appropriate for field use.

We propose to develop a suite of advanced solid state UV light emitters with performance attributes that will significantly advance the capabilities of portable fluorescence based sensor systems. 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 offer higher fluorescence yields and enhanced discrimination for the detection of biological and nuclear materials. We will implement these solid state UV sources into an innovative fluorometer that will use modulation spectroscopy and a 16 channel avalanche photodiode (APD) detector 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 limit the effectiveness of the current generation of compact biosensor systems.

## Accomplishments

The first year goal of this project was to complete the initial design and evaluation of a sensor test bed using “first generation” deep UV LED sources fabricated by our device team at Sandia. Due to the availability of “research grade” commercial 340 nm LEDs, we made a decision to use commercial 340 nm LEDs in our initial sensor test bed development efforts rather than develop 340 nm resonant cavity LEDs (RCLEDs) as originally planned. We shifted our in-house UV light emitter development efforts to the higher risk and more aggressive second year milestones of optimizing LEDs in the 250–280 nm range. We made three significant achievements in the 250–280 nm LED development: (1) demonstration of 2x higher power from 1mm x 1mm interdigitated contact 270–275 nm LEDs through threading dislocation reduction in the AlGaN materials; (2) demonstration of a longer 270–275 nm device lifetime, including > 200 hours for 271 nm devices with 90 microwatt output powers; and (3) development and characterization of LEDs with wavelengths as short as 251.5 nm.

Our biosensor team constructed a setup to evaluate the radio frequency (RF) modulation characteristics of commercial 340 nm LEDs. Initial tests conducted on commercial 340 nm LEDs showed excellent modulation characteristics (> 70 percent modulation depth) in the frequency range (~70 to 100 MHz) required to detect the nicotinamide adenine dinucleotide (NADH) related biosignature, which has a fluorescence lifetime of ~13 ns. We assembled a first generation test bed to test phase-sensitive detection hardware for applicability in biosensors. The test bed consists of a high numerical aperture (n.a.) lens system that images the LED light onto a sample cuvette and another high n.a. lens system that collects (90 degree) scattered and fluorescent light from the sample and images it onto a photomultiplier tube. A high frequency signal generator drives the LED at frequencies up to 2 GHz (although 10s of MHz are more typical) and a radio frequency lock-in amplifier performs phase sensitive detection of the signal from the photomultiplier. Currently, we are using a nonfluorescent reference sample and a biosimulant, *Bacillus thuringiensis*, to characterize and optimize the test bed.

## Refereed

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# SMART Micropreconcentrator for Integrated Preconcentration and Detection of Chemical Agents and Explosives

*79763*

*R. P. Manginell, M. P. Siegal, D. R. Adkins, V. M. Hietala, D. R. Wheeler, E. J. Heller*

## Abstract

In almost any chemical analysis system, there are three important stages: sample preconcentration, sample separation, and sample identification. This project focuses on developing a unique smart preconcentrator (Smart PC) that allows sample collection time to be adjusted on-the-fly to best suit the ambient concentration of target compounds. The device is constructed by placing a thin-film heater and adsorbent layer onto a Sandia developed microelectromechanical system (MEMS) microbalance, known as a pivot plate resonator (PPR). The PPR is set into torsional oscillations by Lorentz forces generated by the interaction of an alternating current through the device and an external transverse magnetic field. The PPR, and thus the Smart PC, actually measures the mass of an analyte as it is collected. For high target analyte concentrations, rapid collection and desorption are performed. For low target concentrations, slower analysis is acceptable. We are developing the Smart PC to combine selective preconcentration and detection functions into a single microscale platform. The Smart PC will provide an early indication of an agent's presence and serve as a front-end trigger to initiate further analysis. A patent application has been submitted for the Smart PC.

## Accomplishments

We completed all milestones for this year. Early in the project we encountered fabrication delays, and we made a slight scheduling change and improved our fabrication process to avoid future delays. This process allows complete KOH etching, complete Bosch etching, or a combination of these approaches. Descriptions of our milestones and accomplishments are provided below.

- *Optimize resonator for Smart PC mass detection:* We made an analytical model of the device that compares very well with experiment. This shows that the beam thickness and magnetic fields have the biggest impact on performance.
- *Fabricate heated resonators with optimized mass detection:* We fabricated and tested resonators; control circuitry is already showing adequate sensitivity.
- *Perform tests on optimized devices:* Tests demonstrated the ability to exactly weigh the amount of analyte.
- *Select high capacity coating:* We will use both sol gels and nanoporous carbon, perhaps in combination.
- *Demonstrate Smart PC concept with rack electronics:* We first used a simple control circuit, then developed a more advanced circuit described below.
- *Develop drive and detection circuits using commercial integrated circuits:* We developed a self-resonating, self-zeroing circuit with digital output proportional to mass adsorbed.
- *Develop integrated μChemLab$^{TM}$ using PPR microbalance:* We built a first generation integrated system.
- *Produce PPR optimized for < 100 picogram mass sensitivity:* We optimized the device that is showing 10–100 pg sensitivity.

# Bead Based Multiplexed, Orthogonal, BioWarfare/Infectious Disease Detection Microsystem and Technologies

*84267*

*P. C. Galambos, M. S. Derzon, C. J. Bourdon, C. D. James, C. S. Yun, K. Rahimian, M. M. Hopkins, J. L. McClain, I. Brener*

## Abstract

Biological weapons agent (BWA) detection is a critical national security need. Current BWA detectors are limited in their ability to handle raw samples containing interfering constituents, such as blood or soil, and in their ability to rapidly detect multiple agents simultaneously. In this project, we will address these limitations by developing the critical components of a magnetic, bar-coded bead based detection microsystem. This year, our efforts to create a multiplexed biodetection platform focused on enabling components for the multipathogen biodetector. We tackled the highest risk issues first. These were: proof-of-principle development of a stable quantum dot (QD) encoded magnetic core bead; obtaining an acceptable operating point for the high flow rate bead trap, taking into account the system issues of temperature rise, back pressure, current, and channel size; and determining the primary system conditions (power, volume, flow, bead detection rate) for a handheld unit.

We fabricated QD magnetic beads in several sizes and colors. They exhibit high signal intensity and were used in prototype microfluidic components. We obtained operating parameters for the magnetic trap in a flat, low flow device configuration and developed both experimental and theoretically consistent operating characteristics. We fabricated and characterized a mesoscale pump that meets the handheld system objectives, in terms of flow rate and pressure. We demonstrated a massively parallel bead focusing concept for two stage optical detection based on an array of dielectrophoretic energy wells. Together, these accomplishments demonstrated feasibility for the highest risk components of the envisioned detection platform. Patent disclosures have been submitted on the bead and fluid handling microsystem.

## Accomplishments

Our FY 2005 accomplishments exceeded our planned milestones. Milestones were met in the QD magnetic bead development, magnetic trap characterization, mixing/hybridization experiments and designs, and optical detector design and initial experimentation. We achieved additional accomplishments in system modeling, development of an integrated mesoscale gear pump (the basic system level microfluidic backbone), and expansion of the bead based concept from BWAs to include CBNE (chemical, biological, nuclear, and explosives) agents.

We successfully demonstrated the three most important milestones in terms of project risk reduction. These were the development of the bead, characterization of the magnetic trap operating characteristics, and fabrication of an integrated microsystem and mesoscaled pump. We created stable QD impregnated magnetic beads, enabling multiplexed detection in an integrated microfluidic format. We incorporated the QDs into commercially available polystyrene matrix, magnetic core beads. We fabricated 1 µm diameter yellow, orange, and green beads of high intensity. This was the project's most critical milestone and proof-of-principle achievement.

The second milestone was fabrication and characterization of the electromagnetic high flow bead trap with respect to flow rates, channel dimensions, and joule heating. The data define an operating phase space in the channel area, current, temperature and flow rate that is acceptable for biological sample processing. We designed, fabricated, and tested a high-throughput mesoscale electromagnetic trap using ceramic firing technology, and a micromachined electromagnetic trap and fluid channel using standard lithography methods.

The third milestone was integrating a pump, a flow channel, a microscope (50X), and a power and light source in a pager sized package. We demonstrated a maximum flow rate of 1.6 ml/min at zero back pressure in this prototype bead handling microfluidic system.

# Terahertz Detectors for Long Wavelength Multi-Spectral Imaging

*84271*

*M. C. Wanke, J. L. Reno, A. D. Grine, C. Highstrete, B. A. Simmons, E. A. Shaner, A. A. Talin, M. Lee*

## Abstract

We are developing an emerging terahertz (THz) detector technology toward the goal of making a fast focal plane array for multispectral imaging in the THz frequency range. Implementation of this technology into an array will enable two-dimensional imaging of objects in the THz frequency spectrum and spectroscopic identification and discrimination of materials. This detector concept is based on a high mobility field effect transistor. The observed photoresponse is narrowband, electrically tunable and has a response time around 0.1 ns, enabling heterodyne operation. FY 2005 goals were aimed at elucidating the physical detection mechanism, raising the operation temperature above 77 K, enhancing the sensitivity, and optimizing size and coupling to single pixel elements.

In addition, we incorporated an effort to employ nanotubes as detectors. Research focuses on studying the high frequency properties of nanotubes to better understand their potential.

## Accomplishments

We improved the performance of the grating-gated field effect THz detectors significantly and made the first measurements on the high frequency properties of carbon nanotubes (CNTs). Both tasks started with overcoming processing challenges. We developed a new etch geometry for the field effect devices to increase the yield and reliability, getting metal contacts and gates to climb over all four walls of a device mesa. For the nanotube devices, we developed coplanar waveguide structures and the very aggressive lithography required to achieve them.

For the grating-gated detectors, we started exploring the device response as a function of detector size. We discovered that single quantum well detectors perform similarly to double well detectors, and demonstrated three orders of magnitude improvement in responsivity using a modified gate bias technique. We also found a photovoltaic signal raising the potential for lower power consumption, observed a photoresponse above 77 K, and measured a low temperature detection bandwidth approaching 10 GHz. All of these are positive achievements towards our goals. We still have not developed the fundamental physical principle on which the device operates, although we have reduced the number of possibilities greatly.

For the CNTs, we successfully demonstrated the first test platform compatible with both directed CNT assembly and broadband high frequency measurements. Using this, we made initial measurements of CNT reflection and transmission characteristics from microwave to millimeter wave frequencies. This first ever high frequency measurement of nanotubes provided promising results, but also indicated improvements that need to be made for conclusive results. We started a redesign to make these improvements.

## Refereed

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M.C. Wanke, M. Lee, E.A. Shaner, J.A. Simmons, and J.L. Reno, “Fast, Tunable, THz Detector Based on a Double-Quantum-Well, Field-Effect Transistor,” presented at the MRS Fall Meeting 2004, Boston, MA, December 2004.

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, “Single-Quantum-Well Grating Gated Terahertz Plasmon Detectors,” submitted to *Applied Physics Letters*.

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, “Plasmon Based Grating Gate Detector,” presented at EP2DS, Albuquerque, NM, July 2005.

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, and J.L. Reno, “Plasmon Based Grating Gate Terahertz Detector,” presented at the APS March Meeting, Los Angeles, CA, March 2005.

## Other Communications

E.A. Shaner, M. Lee, M.C. Wanke, A.D. Grine, J.L. Reno, and S.J. Allen, “Grating Gated FET’s as Narrow-

band, Tunable Terahertz Detectors," presented and in *Proceedings of SPIE 2005*, Orlando, FL, March 2005, vol. 5790, pp. 116–122.

M.C. Wanke, "Electrically Tunable Terahertz Detectors for Spectroscopic Imaging," presented at the Joint Terahertz Imaging System Technology Assessment, Falls Church, VA, September 2005.

# Engineering Sciences

## Coupled Solid-Fluid-Mechanical Computational Modeling of Fracture and Fragmentation in Geomaterials, Such as Hard and Deeply Buried Targets (HDBTs)

*52537*

*R. A. Regueiro, R. E. Jones, R. P. Jensen, D. S. Preece, A. F. Fossum*

### Abstract

This effort addressed modeling of fracture and fragmentation in geomaterials caused by various loading and environmental conditions and required state-of-the-art developments in experimental, constitutive modeling, and computational solution method technology. This project was undertaken in order to develop a predictive capability to model the transition of continuous rock like material to fragmented rock material within the context of coupled solid-fluid-mechanical physics. This capability will support vulnerability models of Hard and Deeply Buried Targets (HDBTs). The project successfully developed computational analysis tools that should be useful in modeling fracture and fragmentation in geomaterials such as concrete, rock, frozen soil, and heavily over-consolidated clay materials that are encountered in foundation construction, tunneling construction, oil and natural gas production, and depleted reservoirs used for subsurface sequestration of greenhouse gases.

### Accomplishments

The project successfully developed computational analysis tools that should be useful in modeling fracture and fragmentation in geomaterials.

(1) An implicit integration scheme was developed for the Sandia GeoModel material constitutive model. The model has been implemented in the Tahoe code for numerical analyses.

(2) A bifurcation analysis of the Sandia GeoModel was undertaken, and a numerical three-dimensional (3-D) bifurcation algorithm was implemented in a finite element code to determine loss of ellipticity of the acoustic tensor.

(3) Elastic-plastic and rigid-plastic cohesive zone models for geomaterials were developed for a post-bifurcation, traction-displacement constitutive law. The model was implemented into the Tahoe code.

(4) An enhanced strain, embedded discontinuity 3-D element implementation was achieved.

(5) A discrete-element/finite-element coupling procedure was established for one-way coupling. Example calculations have demonstrated the capabilities.

### Refereed

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,” *Computational Methods Applied Mechanical Engineering*, vol. 194, pp. 5109–5138, 2005.

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# Ray Model of High Frequency Cavity Scarring

*67064*

*L. K. Warne, M. B. Higgins, H. G. Hudson, J. D. Kotulski, R. E. Jorgenson*

## Abstract

The development of high frequency electromagnetic sources leads to an interest and a need to understand the performance of systems subjected to short wavelength coherent radiation. Interaction with these systems is usually a two step process: (1) radiation enters the system interior (cavity) then (2) couples to interior antennas or circuits. These frequencies are usually beyond what numerical simulations can handle directly. Furthermore, the irregular nature of antenna responses argues for an alternative approach that yields more directly usable information.

In recent years we analyzed these problems using concepts that have been developed in quantum mechanics (the approach to the classical limit). Given that the cavity is irregularly shaped, we assumed the eigenfunctions (modes) to be normally distributed. However, it is known 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.

This project is devoted to exploring the use of 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 would allow better facility designs (mode stirred chambers). It would allow better interpretation of probe measurements and predictions of electromagnetic compatibility, covering the entire range of electromagnetic requirements. Finally, it would provide the underpinning and possible modifications for electromagnetic field level controls in explosive handling facilities (V curve).

## Accomplishments

Work in FY 2004 on two-dimensional cavities identified two issues with the existing time harmonic scar theory that had to be resolved in order to move forward. The first was to resolve the reason that the asymptotic theory for large radius of curvature on convex boundaries seemed to work better than expected for practical cases where the radius of curvature was not large. The second was to find a way to generalize the theory to concave boundaries. These two issues were both resolved by generalizing the time harmonic scar theory to curved ray trajectories.

A high frequency method that was used for describing stable modes along curved trajectories in laser cavities was the starting point for this approach. We applied the random phase boundary conditions from the unstable scar theory to this approach and constructed solutions for the unstable modes supported by convex boundaries, which did not require as stringent a restriction on radius of curvature as the existing theory. This explained why the existing theory yielded accurate results for smaller radii of curvature of the convex surface.

Next, in the concave geometry, we generalized the theory to multiple regions about the interior foci (which are unique for this situation). A method of matching was devised using a sub-wavelength shift in focal point from the geometrical position. The results for the scarred mode spatial distribution and for the modal amplitude statistics agreed with two-dimensional numerical experiments on the stadium cavity.

In both applications, the normalization condition for the scarred modes was found by applying the electromagnetic energy theorem. This was required because of the variable nature of the eigenfunction amplitude along the scar in this more accurate curved treatment (it was essential for the multi-region treatment of the concave boundary). A journal paper is being prepared on the two-dimensional scar theory.

Finally, a three-dimensional ray tracing algorithm was set up to enable the determination of modal frequencies and stability exponents in three dimensional cavities. The three dimensional generalization of the modal scar theory for convex and concave geometries

will be completed in FY 2006 along with a limited set of experimental measurements.

FY 2005 accomplishments:

(1) Generalized the time harmonic theory of unstable periodic orbits with convex boundaries to elliptical coordinates to estimate accuracy and resolve discrepancies in stability exponents.

(2) Found a means to apply the theory of unstable periodic orbits to concave boundaries with interior foci. This involved a treatment using multiple interior regions along the scar orbit.

(3) Found a way to normalize the eigenfunction in the multi-region scar by use of the electromagnetic energy theorem.

(4) Demonstrated spatial variations along the multi-region scars to match the simulations and explain the behavior in each region. The scar wavefunction statistics captured the variations exhibited in the simulations.

(5) Set up a three-dimensional ray tracing algorithm.

# Noncontact Surface Thermometry for Microsystems

*67067*

*S. P. Kearney, J. R. Serrano, L. M. Phinney*

## Abstract

Future systems at Sandia will include microelectromechanical systems (MEMS) components playing critical roles. The Discriminating Micro-Switch (DMS), nonvolatile memory, vertical cavity surface emitting lasers (VCSELs), and micro-sensors are currently being designed and evaluated for near term applications. Characterization of thermal energy transport in these devices is necessary for understanding their performance in normal environments and their response to abnormal environments. Although quantitative temperature measurements are essential for implementation of MEMS technology into engineered systems, Sandia's capability in this area is significantly lacking.

We will cultivate state-of-the-art diagnostics for microscale surface temperature measurements and apply them to Sandia MEMS components. These diagnostics will be non-contact in nature, offer submicron resolution, minimize indirect heating, and apply to most materials. Micro-thermoreflectance and micro-surface Raman scattering are complementary techniques that meet these criteria. Both methods utilize a low-power probe laser of arbitrary wavelength. Thermoreflectance methods utilize the pre-calibrated temperature dependence of the surface reflectivity of MEMS structures. Thermoreflectance methods have great potential for measurements on metals and highly reflective surfaces, but require extensive calibration and suffer from weak signal strength. Raman methods take advantage of the temperature-sensitive spectrum of inelastically scattered probe-laser light. They are more complex than thermoreflectance techniques but also more powerful. Raman methods are effective on absorbing surfaces, can potentially reduce the surface calibration need, and yield information regarding lattice structure and state of stress that are useful for thermophysical model development.

This work will facilitate communication between diagnostics development teams, MEMS designers, and systems engineers. The project will provide valuable experimental temperature diagnostics for MEMS component testing and certification and for model development and validation.

## Accomplishments

Principal achievements during FY 2005 include the following:

- Use of Raman calibration spectra to probe Joule-heated thermal actuators and polysilicon under steady-state conditions
- Quantification of the uncertainty in steady-state Raman temperature measurements of +/-10 K
- Fabrication of a controlled bending apparatus for temperature-stress calibrations
- Completion of a prototype thermoreflectance thermometry instrument
- Acquisition of temperature-dependent reflectivity data from crystalline silicon and Sandia Ultraplanar Multilevel MEMS Technology V (SUMMiT V™) polysilicon films
- Steady-state Raman thermometry of a heated atomic force microscope (AFM) probe by the Georgia Institute of Technology (Georgia Tech)
- Preliminary joint measurements of temperature and stress
- Early development of a state-of-the-art surface enhanced Raman scattering (SERS) and filtered Raman scattering instruments by Sandia and Georgia Tech researchers.

We used Raman to obtain steady-state temperature maps from working Joule-heated microscale actuators, which are being considered by Sandia for several active MEMS applications for Defense Program systems. These are the first ever spatially resolved quantitative temperature maps from thermal actuators, where the small 3 micron width of the devices had previously been unresolved. These data have been used to validate a thermal model developed under a previous LDRD project. Simultaneously, researchers at Georgia Tech, under the sponsorship of this LDRD project, performed the first ever temperature mapping of atomic force microscope tips under working conditions.

Raman peak positions in semiconductor materials have been also been observed to be sensitive to the material's state of stress. This can cause errors in temperature measurements, but it can also be exploited for simultaneous mapping of temperature and stress in working MEMS. We made preliminary measurements

of temperature and stress by utilizing the stress-independent line width of the Raman transitions as an indicator of temperature and the Raman peak position as a stress sensitive probe. We performed these measurements on both constrained micro- and Comtois-thermal actuators. We are currently pursuing methods to improve the accuracy of the stress measurement using in-house fabricated mechanical testing facilities that have been specially tailored for micro-Raman probing.

Sandia and Georgia Tech researchers have been working jointly to improve the temporal resolution of Raman temperature mapping via a state-of-the-art SERS technique. This method utilizes a sharp, nanometer-scale metal tip in the vicinity of the Raman-probed surface. The close proximity of the two surfaces and the probe laser beam excites surface plasmon resonances in the metal that can enhance the strength of the Raman-scattered electric field by several orders of magnitude. The signal enhancement can be used to reduce the required integration time of the Raman signal and could make more time-resolved measurements feasible. We are also investigating a filtered Raman technique in which a fine-scale spectral filter replaces the need for spectroscopic data in conjunction with SERS for further enhancements in temporal resolution. Early theoretical calculations show that the filtered Raman technique is feasible, and demonstration of this diagnostic will be a major technological advance.

We are also pursuing microscale thermoreflectance as a temporally resolved temperature diagnostic tool for MEMS. This method exploits small but detectable temperature-dependent changes in the reflectivity of a material for relative temperature measurements. The technique does not require spectroscopic measurements so that data acquisition rates are much faster than Raman probing. We acquired accurate reflectance calibrations of SUMMIT V™ polysilicon films and will use these data in FY 2006 to obtain relative temperature maps.

## Refereed

S.P. Kearney, L.M. Phinney, and M.S. Baker, “Spatially Resolved Temperature Mapping of Electro-Thermal Actuators by Surface Raman Scattering,” to be published in the *Journal of Microelectromechanical Systems*.

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# High Speed Interferometric Deformation Measurements

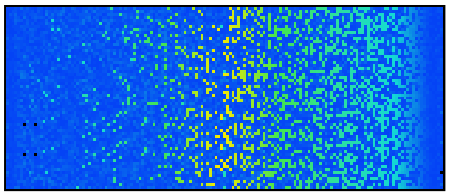
*67068*

*P. L. Reu, G. C. Stoker, J. E. Massad, B. D. Hansche, D. O. Smallwood*

## Abstract

There are a number of converging factors that are driving the need for non-contact dynamic measurements. These include the development and validation of detailed structural models that need experimental data for comparison and the design and testing of microelectromechanical systems (MEMS) with parts such as gears and accelerometer reaction masses that exhibit dynamic motion. Wide area measurement of out-of-plane motion has proven difficult, leaving a strong need for a non-contact measurement technique that provides quantitative data on a dense spatial grid, a velocity "image."

This project involves the significant extension of laser Doppler velocimetry (LDV) techniques from a single point method into a widefield measurement. Typically, an LDV uses a probe beam focused to a single point, and the returned Doppler shifted beam is interferometrically measured, resulting in a heterodyned difference frequency proportional to the object velocity. For typical structural mechanics velocities, this frequency is in the tens of megahertz, requiring the use of fast diode detectors. This limits the technique to single point measurements, with velocity images built up by scanning the probe beam. This can take several minutes for a velocity image of an object in steady state vibration and is impossible for transient events. This project extends this measurement into a parallel detection system, measuring thousands of velocities simultaneously. This is accomplished by using illumination modulation to heterodyne the signal down to frequencies at which a conventional video system can directly respond to the Doppler frequencies. Tracking of the Doppler frequency for control of the heterodyning frequency, for camera demodulation purposes, is accomplished via a standard single point LDV positioned in the image. In addition to modulation of the signal, Joint Time Frequency Analysis (JTFA) techniques are used with sub-Nyquist sampling to significantly expand the bandwidth of the video system and increase the velocity bandwidth detectable.



*Velocity movie of rotating block.*

## Accomplishments

Building on the successful proof of concept experiments conducted in FY 2004, we assembled and tested a prototype benchtop widefield LDV system. We explored microchannel plate intensifier gating for use as both modulating and optical anti-aliasing hardware. Due to brightness limitations of the output phosphor and slower than desired response times, current intensifiers will not work for this application. As an alternate method, we added an acousto-optic modulator (AOM) to the system for amplitude modulation of the illumination laser beam. The laser modulation allows the Doppler signal (proportional to the velocity) to be heterodyned down to camera frame rate frequencies (typically 6 to 76 thousand frames per second depending on field-of-view).

To test the widefield LDV, we used a rotating and accelerating plate that provided a controlled time and spatially varying velocity field to the system. We successfully demonstrated the modulation technique by measuring a range of velocities (i.e., different modulation frequencies) at different locations on the plate. We utilized JTFA techniques to convert single pixel intensities to time varying velocity information. Due to camera hardware, bandwidth limitations are still a problem for this technique. Sub-Nyquist sampling (i.e., below the Nyquist rate of the highest frequency) and alias unwrapping techniques that we are exploring show promise for extending the effective bandwidth of the system. We automated basic image processing methods to unwrap the aliased frequencies and process the velocity images. Using these newly developed routines, we successfully created a spatial and time varying velocity movie of the rotating plate.

We added a single point LDV system to the widefield LDV to determine the optimum AOM modulation

frequency. The velocity at this point is measured by the computer, and an appropriate modulation frequency is calculated and applied to the AOM. This has the benefit of setting the Doppler frequency at the LDV point to zero hertz (or any chosen offset). This removes the temporal aliasing problem by allowing the target velocity to be tracked and maintained within the bandwidth of the camera. Using the tracking method, we successfully conducted tests utilizing a pendulum.

We are investigating additional work on fundamental topics in LDV systems surrounding laser speckle attributes, including speckle fringes, speckle size, and speckle motion. We experimentally investigated optimization of the number of speckles per pixel and generated interesting directions for further research. Computer simulations to help understand speckle phenomenon are ongoing. A patent application for the widefield LDV is in the final stages of preparation before submission.

# Fundamentals of Nanofluidics

*67069*

*R. H. Nilson, S. K. Griffiths*

## Abstract

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, while molecular scale simulations alone cannot address the submicron to micron scale domains of interest in most applications.

In this project we are developing multiscale models of flow and transport within submicron channels by combining Density Functional Theory (DFT) with classical continuum mechanics. DFT is being used to compute structured fluid and ion density profiles based on Lennard-Jones, Coulombic, and hard sphere 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.

In FY 2005 we verified our modeling by comparing our DFT-based predictions of fluid velocities with published molecular dynamics (MD) simulations of shear-driven Couette flows and electroosmotic flows in channels ranging from 1 to 20 nm. In addition, we developed a two-dimensional DFT model by extension of the one-dimensional model developed in FY 2004. Our comprehensive DFT-based modeling of electroosmotic flows demonstrated that classical continuum modeling may overestimate fluid velocities in nanochannels by an order of magnitude.

The multiscale models under development are needed to design advanced microChemLab and electrokinetic pumping devices. Additional applications include lubrication of microfabricated devices for weapon arming and safing, hydrogen storage in nanostructured materials, thin film flows in heat-pipes, and microscale injection molding. Finally, the development of physics-based constitutive relations describing nanofluidic phenomena represents a fundamental and enduring contribution to the field of fluid mechanics.

## Accomplishments

Our goal is to develop multiscale models of flow in submicron channels by combining classical continuum mechanics with DFT modeling of the non-continuum physics operative within molecular layers adjacent to solid surfaces. This approach enables incorporation of nanoscale physics into continuum codes in a manner that is fundamental and yet far more practical than the alternative of direct MD simulation. In FY 2004 we developed a coupled DFT/Continuum model based on preliminary one-dimensional models of essential physics.

FY 2005 activities focused around: (1) development and testing of constitutive models describing fluid viscosity and surface slip, and (2) investigation of differences between classical fluid mechanics and nanofluidics. Another major advance this year has been development of a two-dimensional version of the one-dimensional DFT model that was initiated last year. An overview of FY 2005 accomplishments is given below.

We performed a series of calculations to evaluate the accuracy of a modified Chapman-Enskog viscosity model based on averaging our computed DFT density profiles over a surrounding sphere of fluid. We found results obtained by this method to be in excellent agreement with published MD simulations of Couette flow in the gap between one stationary wall and one moving wall. These MD results and our DFT calculations show nearly identical increases in viscosity as the channel size decreases, owing mainly to the attractive forces and confinement by the walls. We also found our DFT-based simulations of electroosmotic flow in nanoscale channels to be in good agreement with the MD simulations conducted by others. The advantage of DFT over MD is that computer times are reduced by orders of magnitude.

We began formulation and testing of a physics-based model of surface slip. The slip velocity is deduced by performing a momentum balance on the monolayer of fluid molecules adjacent to the solid surface. The shear force on the fluid side of this layer is computed from the velocity gradient and the local viscosity in the usual manner. However, the momentum exchange between the monolayer and the wall is computed by statistical modeling of molecular collisions

with the stationary wall molecules. Initial comparisons with published MD simulations appear encouraging, but further progress requires multi-dimensional DFT modeling of the fluid density field. To meet that need, we are now using a newly developed two-dimensional DFT model to determine local fluid densities and collision rates at different locations along the structured solid surface.

A large number of calculations were performed to fully explore the departures of electroosmotic flows from classical continuum behavior. We chose to focus on these flows because electric forces, rather than pressure forces, are a preferred means for driving micro- and nano-flows. Our DFT model predicts smaller flow speeds and smaller zeta potentials than the classical Poisson-Boltzmann theory, particularly when channel sizes are small and surface charge is large. Both of these features are also seen in the few MD simulations that appear in the literature. However, our DFT approach facilitates parameter studies spanning thousands of cases, rather than a few.

We explored the efficiency of electrokinetic pumps based on nanoscale channels. We found that optimized pump designs always involve very small channels having dimensions on the order of the Debye thickness (< 20 nm).

## Refereed

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R.H. Nilson and S.K. Griffiths, “Modeling Nanoscale Flows by Combining Density Functional Theory with Classical Continuum Mechanics,” presented at the 8th 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, D. J. Holdych, M. J. Stevens*

## Abstract

Self-assembly of biomolecules is a promising avenue toward the development of new materials. Engineering principles for micro- and nano-fabrication can be discovered from the study of molecular self-assembly phenomena in nature. Biological tissues serve as model factories for the production of macromolecular structures, where the processes of molecular self-assembly and macroscopic growth are integrated in robust systems that display self-healing and adaptivity. Growth occurs as nutrients, amino acids, and enzymes undergo mass transport and react to increase tissue mass. At the molecular scale, there is considerable understanding of the reactions for self-assembly of tissue components such as collagen. There is less understanding of the thermodynamic driving forces and of how these interact with growth processes to form multicellular tissue. These processes are also influenced by mechanics at the continuum scale.

The project will study the processes of self-assembly and growth in collagen from the macromolecular to the continuum scale. The mathematical description of growth is a highly complex system that requires mixture theory with coupled diffusion-reaction. This system also requires mechanics that differ significantly from classical continuum formulations of closed systems. The models will be developed and validated with collaborators at the University of Michigan, where they have the capability for growing in-vitro tendon constructs and characterizing their microstructure and response. This project provides the opportunity to uncover fundamental principles in the extension of characteristics of biological systems to the development of new engineering materials. Further, it provides the opportunity for Sandia to extend its leadership in computational and engineering science to a pioneering role in biotechnology.

## Accomplishments

In FY 2005, we extended the coupled mechanics-transport formulation to include (a) Michaelis-Menten enzyme kinetics in the source term for collagen production, (b) reformulation of the problem in the current configuration to enable the application of physically-relevant boundary conditions on concentrations, and (c) introduction of chemical species transport by fluid advection and chemical potential gradients. Presently, we are implementing spatial stabilization schemes for the transport equations. We have used the model and the code to elucidate the following aspects of coupled mechanics and transport in biological tissues:

(1) Fickian behavior is possible only in tissue that is unsaturated by the fluid. However, if the tissue is saturated, concentration gradient-driven transport is still possible through the stress gradient since the stress is concentration-dependent.

(2) Only saturated tissues swell due to fluid infusion. Unsaturated tissues have the capacity to absorb some fluid without swelling.

(3) The introduction of voids must accompany tensile stretching of the tissue, since fluid transport is not instantaneous.

Through numerical simulations we demonstrated stress gradient-driven “pumping” of fluid through the tendon construct. Enzyme kinetics-driven source terms can now be incorporated with this computation to study the influence of cyclic loading on growth. In other simulations, we obtained the increase in mass and volume of the tendon construct due to growth. Diffusion-dominated and reaction-dominated regimes of growth were identified. The stiffness and strength increase of the tendon after growth was demonstrated by a computation of the tendon loaded in uniaxial tension before and after growth.

At the mesoscale level, we developed a finite-difference (FD) model to simulate nutrient transport in a fiber, using the flow field obtained from lattice-Boltzmann (LB) simulations. The FD model makes use of the LB square grid, allowing a standard centered difference stencil to be used in the bulk of the fluid. In the vicinity of the curved fibril surfaces, we used the no-flux boundary condition to eliminate dependencies of the original centered difference stencil on nodes outside the fluid domain. This general approach for modifying the difference stencil admits a range of modified stencils, depending on exactly how the boundary con-

dition is applied. At the same time, there exists an established stability criterion for the difference stencil. Therefore, we posed the stencil modification process as a constrained optimization problem, with the stability criterion as the constraint and a proposed error norm as the objective function. This problem can be solved with available routines but becomes exceedingly expensive when applied to three-dimensional simulations. Therefore, we formulated the final FD model to directly construct a modified stencil that satisfies the stability criterion. The model then takes one step in the direction of minimizing the proposed error norm. Benchmark results compare well with those from other methods proposed in the literature. Solute transport through a random arrangement of cylinders was also simulated. We will compare these results with future simulations using the mixture theory model.

Finally, at the macromolecular level, we performed atomistic simulations of collagen to study the strength of the molecule at the atomic scale. We performed simulations in both a tensile pull and perpendicular tearing mode. We obtained force versus distance data. We are now working on correlating these data with the structural and energetic transitions. We now understand that hydrogen bonds are not the main interaction holding the collagen triple helix together.

# Investigation of Liquid Jet Break-Up and Dispersion

*79764*

*R. A. Jepsen, S. S. Yoon, T. J. O'Hern*

## Abstract

Initial studies show the importance of air in causing the splashing phenomenon and the subsequent finger formation for a large scale liquid droplet impact. While previous work focused on the experimental aspect and addressed the issues associated with the Rayleigh-Taylor instability, additional experimental investigation and the relevant computational modeling have been performed to obtain additional insight on the large scale splashing phenomenon. Previous modeling efforts did not consider the effect of air by starting the simulation at the time of droplet-liquid contact with the substrate. Here, we start the simulation using the VOF (volume of fluid) method at a location one diameter upstream so that the compressed air effect, due to a falling droplet, is properly taken into account. Both the experiments and simulations demonstrate that the displaced air obtains momentum from a falling droplet and induces a vortex motion right above the contact surface. The splashing (or ejection) occurs when the initial edge of the impacting and spreading liquid is entrained into the displaced and accelerated air. It is also hypothesized that the perturbation generated during the splashing process is radially propagated and is the fundamental instability that eventually forms fingers at the rim of the spreading liquid.

## Accomplishments

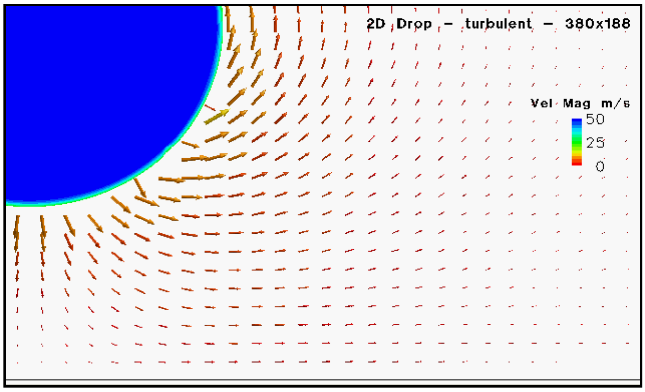
This project has been extremely successful in the last year. All of the milestones for FY 2005 listed below have been met or surpassed, or are ahead of schedule.

(1) Design and construct appropriate fixtures to deliver liquid slugs from actuator sleds.

(2) Make minor modifications to actuator facility to provide enclosure for impact area.

(3) Design diagnostic setup to measure break-up and dispersal processes including phase Doppler particle analysis, high-speed photometrics and track-eye analysis, and load time history of impact using a quartz load cell.

(4) Conduct initial testing and make preliminary diagnostic measurements.




*Movie of computational fluid dynamics.*

We successfully implemented novel experimental techniques and utilized them as originally proposed. Results from low speed (< 3 m/s) or Weber number (< 10,000) impact testing match well with previously published data. In addition, higher speed testing revealed important new phenomena in the impact and splashing characteristics. This work already resulted in one conference presentation and paper, submission of two journal articles, and two additional articles in progress.

This work provides 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. This new research is the foundation that will revolutionize the current theory on dispersion processes with applications beyond our scope, such as ink jet printing and fuel injection combustion.

## Refereed

S.S. Yoon, R.A. Jepsen, M.R. Nissen, and T.J. O'Hern, "Experimental Investigation on Splashing and Finger-like Instability of Large Water Droplets," in *Proceedings of the ILASS Americas, 18th Annual Conference on Liquid Atomization and Spray Systems*, 2005, CD-ROM.

# Accelerating DSMC Data Extraction via Signal Processing

*79765*

*E. S. Piekos, M. A. Gallis*

## Abstract

In many direct simulation Monte Carlo (DSMC) simulations, the majority of computation time is consumed after the flowfield reaches a steady state. This situation occurs when the desired output quantities are small compared to the background fluctuations. For example, gas flows in many microelectromechanical systems (MEMS) have mean speeds more than two orders of magnitude smaller than the thermal speeds of the molecules themselves. The current solution to this problem is to collect sufficient samples to achieve the desired resolution. 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 samples to cut the error in half. The proposed work is intended to improve this situation by employing more advanced techniques from fields other than solely statistics for determining the output quantities.

Our strategy centers on exploiting information neglected by current techniques that collect moments in each cell without regard to one another, values in neighboring cells, or their evolution in time. Unlike many previous acceleration techniques that modify the method itself, the proposed techniques are strictly post-process so they may be applied to any DSMC code without affecting its fidelity or generality. Many potential methods are drawn from successful applications in a diverse range of areas, from ultrasound imaging to financial market analysis.

The potential gains of this research are considerable. Previous work in the spatial filtering category, using ideas from large eddy simulations, showed more than an order of magnitude reduction in compute time. The proposed program can impact research and analysis capabilities in all areas where DSMC has been applied, which range from thermal transport in silicon films to gas damping in MEMS to neutron tube modeling to the Columbia accident investigation.

## Accomplishments

We evaluated three methods for exploiting spatial information in detail this fiscal year: nonrecursive digital filters, Fourier filters, and iterative reduction of noise (IRON). In addition, we performed a study to determine the optimum sampling rate for steady-state calculations.

The nonrecursive digital filters examined in this work may be viewed as “moving window averages,” where a data point is replaced with a weighted average of itself and a select group of its neighbors. The differences between the filters are simply the size of the group and the weighting scheme.

Of the seven nonrecursive filters evaluated, the 11 point modified quadratic filter showed the best performance overall. For the collision fraction, the root mean square (RMS) error of the filtered result was 2.1 times lower than that of the unfiltered result at 10,000 samples. Stated another way, the filtered data reach the unfiltered data’s errors at 300,000 samples after only 70,000 samples, a factor of 4.3 fewer iterations. Excluding the time required to reach steady state in the DSMC simulation and to calculate the filter, which is negligible, this factor represents the central processing unit (CPU) time saved by filtering.

Fourier filters were the second type of smoothing technique examined in this project. Under this technique, a discrete Fourier transform is computed using the DSMC data points. This series is then truncated, discarding the high frequency modes assumed to be noise, and the inverse transform is computed. While each data point had a limited domain of influence under the nonrecursive digital filters, the Fourier filter essentially creates a global fit. Choosing the truncation point, therefore, involves a balance between accurate representation of the data and rejection of noise.

We demonstrated that the 32 mode Fourier filter performs slightly better than the nonrecursive filter on both collision fraction and temperature. We observed that the Fourier filter output is generally smoother and closer to the true value than the nonrecursive filter output. Because it is a global fit, the Fourier result is also somewhat less sensitive to localized peaks in the data that, in this case, are nonphysical.

The final spatially-based extraction method evaluated to date is Fippel and Nüsslin’s IRON technique. In this technique, developed to smooth Monte Carlo calculated dose distributions for radiation therapy, the data points are shifted by an optimization routine. The opti-

mization seeks to minimize local second derivatives while also minimizing the motion of each data point.

This technique performed significantly better than its predecessors. In this case, the improvement at 10,000 samples over the unprocessed case is a factor of more than three for the collision fraction and two for the temperature. This technique performed extremely well for the flow speed, which should be zero, improving the error by more than a factor of four. In terms of the number of iterations to reach a given error level, the IRON technique's error drops below the 300,000 sample unfiltered result after only approximately 25,000 samples for the flow speed, 32,000 samples for the collision fraction, and 119,000 samples for the temperature.

In the sampling optimization study, we performed a parameter sweep on the Fourier problem using one-dimensional and two-dimensional codes. We observed that the error is minimized when sampling takes place every 20–25 times the number of moves to cross a cell. We also observed that both codes ran most efficiently, i.e., executed more moves per hour, when using the optimum sampling rate. This acceleration can be attributed to the contribution of an accurate collision frequency to the speed of the code. If the collision frequency is calculated properly, the code will follow an optimum path toward steady state. In total, the difference between the sampling after every move and 25 times the cross cell time may reduce the error bar by a factor of two, reducing overall run time by a factor of four.

# Modal Analysis of Almost Linear Structures

*79766*

*D. J. Segalman, D. Griffith, M. D. Jew, J. P. Lauffer*

## Abstract

Modal analysis has been extraordinarily valuable in the field of linear structural dynamics for structural identification, model validation, model reduction, model synthesis, and uncertainty quantification. Perhaps the greatest value of modal analysis has been in understanding the dynamic properties of structures. Now that we have extended our numerical techniques to structures that admit modest nonlinearities, the mathematics that underlies modal analysis no longer appears to be applicable. Experimental investigations of nonlinear structural dynamic systems show that practically all real mechanical systems display modal-like behavior; therefore, it would be valuable to extend the mathematics of modal analysis to mildly nonlinear systems.

Some mathematical tools have been developed for special types of nonlinearly elastic structures (nonlinear normal modes). However, no useful tools have been developed for problems having intrinsic nonlinearities of the sort that interest us, particularly those having the nonlinear dissipation and softening effects associated with joints. A framework for interpreting and understanding the modal-like behavior of such structures needs to be developed. This project explores several approaches to devising and validating modal-like tools appropriate to such problems.

A major advance was made in identifying why conventional Galerkin methods fail on such problems. That realization led to the development of an augmented basis system that captures the kinematics much better and yields far better analytic results.

Decomposition of mildly nonlinear response systems using spatial filtering allows tracking of system nonlinearities as a function of time and amplitude. More traditional Gabor transform and wavelet methods have limited frequency resolution capabilities and no ability to track damping changes. By combining spatial filtering with time domain parameter estimation techniques, damping estimates and improved frequency estimates, as a function of time and amplitude, can be obtained.

## Accomplishments

- *Computational Goal: Development of at least one mathematical formalism for nonlinear modal analysis.*

We developed a Galerkin method of model reduction using the models of linear system that have the same tangent stiffness matrix as the nonlinear system of interest. Convergence was extraordinarily poor, manifesting a Gibbs-like phenomenon.

Further investigations of the singular value decompositions of the full nonlinear solutions suggested that the system of basis functions needed to be augmented by functions that have discontinuities at the locations of nonlinearity. This important observation led to an entirely different formulation.

The new formulation involves the systematic definition of discontinuous basis functions, exactly suitable to the problems at hand. Behavior of basis systems augmented by these functions is very promising.

- *Experimental Goal: Develop an analysis approach for decomposition of modal-like experimental data.*

We reviewed several approaches for assessing the adequacy of the analytical approach for modeling the mildly non-linear response of structures to track changes in resonance frequencies and damping factors. Conventional approaches like wavelets and short time fast Fourier transforms do not provide sufficient frequency resolution. The approach is to decompose structural response into single mode responses using modal filtering. The assumption is that the non-linearities affect the damping and frequency, but the mode shapes are largely unaffected. The resulting single mode responses are then analyzed using time slices during free decay to estimate damping and frequency. Adequacy of the filtering process is assessed by observing the Fourier transform of the single mode response. The Hilbert transform of the decayed response is used to set error bounds on the damping estimate and to determine time intervals over which this type of analysis is valid.

We applied this approach to simple linear and nonlinear analytical models. Additionally, we used this

approach on data measured by a simple quasi-linear test specimen.

## Other Communications

D.J. Segalman and D.T. Griffith, “Strategies for Model Reduction of Structures with Localized Nonlinearities,” presented at the Sixth Biennial Tri-Laboratory Engineering Conference, Monterey, CA, September 2005.

# Nano-/Micro-Engineered Interfaces for Improved Performance and Reliability

*79767*

*E. D. Reedy Jr., T. D. Nguyen, J. A. Zimmerman, N. R. Moody*

## Abstract

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 continuum finite element simulations. The finite element simulations will investigate how a growing crack is deflected by local heterogeneities to generate a torturous, energy-absorbing crack trajectory. The experimental component of this project will study well-defined patterns of nano-/microscale heterogeneities. Lithographic 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. Interfacial toughness will also be measured with stressed metal overlayers. The experimental results will help guide and validate the modeling effort. The fundamental understanding developed during this study will enable the development of nano-/micro-engineered interfaces with optimized toughness for critical bonded, coated, laminated, and encapsulated defense program components, including those using microelectromechanical systems (MEMS) and emerging nanoscale technologies.

## Accomplishments

We developed and applied atomistic and finite element techniques to model interfacial separation. We also made progress in developing test techniques to measure the toughness of patterned interfaces.

The finite element modeling effort is currently focused on a rippled interface. This geometry allows us to investigate the effect of ripple wavelength and magnitude on interfacial crack propagation. Our most important finding is that finite element simulations do predict a significant increase in the apparent toughness of patterned tungsten/silicon interfaces, like those we plan to test. The calculations indicate that the apparent fracture toughness is twice that of the intrinsic interfacial toughness when there is a 25 nm ripple.

The atomistic simulations concentrated on developing atomistic models for incoherent interfaces of regular geometry. We created a model representative of realistic incoherent bimaterial interfaces by equilibrating a <100> oriented block of gold on top of a <110> oriented block of gold using the modified embedded atom method. We performed both tensile and shear simulations. Much of our effort focused on determining the effect of atomistic system size on calculated results. We determined that relatively small atomistic systems are sufficient to determine the work needed to create interfacial failure.

Our experimental work focused on developing techniques to measure the effect of interfacial topography on interfacial toughness and on creating interfaces with well-defined surface patterns of controlled shape and dimension to test. One success was the adaptation of the four-point bend tests to nanoscale film fracture. We performed this work in close collaboration with a team member at the University of Minnesota. We also investigated several patterning techniques. We found that nanoimprint lithography can create well-formed 200 nm wavelength sinusoidal patterns, an ideal geometry for our initial tests of patterned interfaces.

# 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*

## Abstract

The goal of this project is to provide the critical computational analysis and design tools for photonic band gap (PBG) structures, so they can be built and installed on military platforms without resorting to excessive experimental prototyping. PBG structures inhibit photon propagation over a range of frequencies. For example, PBG structures coated on a hot body can modify the infrared radiation of the body and mask it from detection. PBG structures are created by replicating submicron-sized unit cells on a periodic lattice. By introducing defects in the PBG lattice, the optical band gap can be modified, leading to the possibility of new optical devices.

From an analysis perspective, PBG structures are similar to frequency selective surfaces (FSSs) and phased array antennas in the microwave domain. We have successfully applied EIGER, an electromagnetics code, to analyze how light scatters from infinite planar PBG structures. However, EIGER assumes perfect periodicity, which severely limits its application to realistic problems. We are developing novel computational techniques that will allow us to analyze the effects of lattice truncation, and seams and lattice defects on the scattering characteristics of the PBG structure. The new techniques will also apply to FSS analysis.

This project will significantly expand the ability for Sandia to model PBG structures in real-world applications and will place us in a unique position to meet the modeling needs of Sandia, as well as the military and other customers.

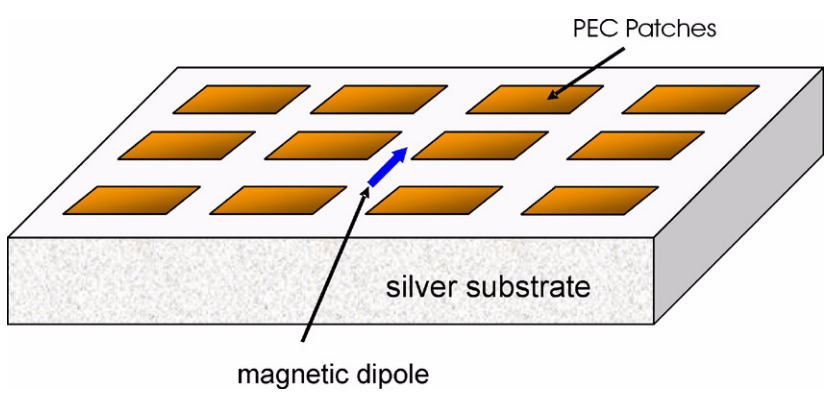
## Accomplishments

We first down selected the techniques that would be applied to analyze the defect and seam. We decided to explore a method known as Green's Function Interpolation with Fast Fourier Transform (GIFFT) to see if it could be used to analyze defects. For the seams, we looked into a method known as the Truncated Floquet Wave Diffraction Theory, which had been used to analyze truncated phased array antennas.

To assist us, we brought Professor Donald Wilton from the University of Houston to Sandia on a three month sabbatical to work on this project. We invited Ben Fasenfest from Lawrence Livermore National Laboratory to Sandia to discuss GIFFT and how it could be modified to analyze defects. We also brought Professor Filippo Capolino to Sandia to explain his work on truncated phased arrays and how this technique could be applied to analysis of seams.

We modified the GIFFT method to analyze defects. We obtained results showing the effect of a simple defect (45 degree rotated dipole in an array of horizontal dipoles) on the dipole current distribution and the far-field of the array. We also applied GIFFT to more complicated structures to study the effect of plasmons on the transmission characteristics of light through a small aperture.

We incorporated the capability to analyze a one-dimensional seam (defined to be a seam between two




*Geometry used in a study of plasmons. Patches are placed periodically on a silver substrate. The magnetic dipole models an aperture through the substrate.*

different, two-dimensional semi-infinite arrays of strips) into EIGER. Finally, we wrote software to compute the Green's function of a one-dimensional array of points for one of next year's tasks.

## Refereed

L.I. Basilio, B. Fasenfest, D.R. Wilton, and F. Capolino, "Efficient Numerical Modeling of Truncation Effects and Defects in Finite Periodic Structures," in *Proceedings of the 2005 IEEE AP-S International Symposium and USNC/URSI National Radio Science Meeting*, 2005, CD-ROM.

L.I. Basilio, W.A. Johnson, D.R. Jackson, and D.R. Wilton, "Numerical Modeling of Finite-Size Plasmon Structures with Enhanced Optical Transmission Using EIGER," in *Proceedings of the Joint 9th International Conference on Electromagnetics in Advanced Applications ICEAA 2005 and the 11th European Electromagnetic Structures Conference EESC*, 2005, vol. 1, pp. 877–880.

# Shock Induced Explosive Chemistry in a Deterministic Sample Configuration

*79769*

*W. M. Trott, A. S. Tappan, J. N. Castaneda, M. R. Baer*

## Abstract

Despite decades of active research, the current understanding of reactive flow in solid explosives still relies on the following: (1) experiments that typically access average behavior in highly specific sample configurations, and (2) models that are purely phenomenological. In heterogeneous energetic materials, shock sensitivity of initiation and sustained reaction is intimately tied to processes occurring at the grain/crystal level. Shock wave fields are three-dimensional and unsteady, arising first at contact points, then coalescing to produce a complex distribution of thermal and mechanical states that are, in turn, highly coupled to fundamental chemical processes of the energetic material. These processes are driven by the highly localized temperature and stress states of the material. The size, intensity, distribution, and coalescence of resulting "hot spots" within the shock-loaded material are considered to be controlling factors in reaction initiation and growth. Unfortunately, in typical heterogeneous formulations (pressed, granular explosive with or without added binder materials), "hot spot" formation and growth is highly stochastic. The complex statistics are a significant barrier to identification and analysis of the important underlying chemistry.

We evaluated different approaches to explore the feasibility of preparing and testing explosive samples in a highly deterministic configuration. The intent is to preserve the essential phenomenology of heterogeneous explosive initiation (a wide range of stress and temperature with extreme conditions localized at contact points and interfaces) while largely constraining stochastic behavior. Transmitted wave profile measurements from shock-loaded samples with a designed deterministic geometry have been performed using both a single-point velocity interferometry system for any reflector (VISAR) and a line-imaging optically recording velocity interferometer system. These designs include (1) a close-packed bed of inert tin spheres with sensitized nitromethane occupying the open volume, and (2) nitromethane or sensitized nitromethane occupying an open volume matrix well-defined by a Robocast sintered alumina insert. We evaluated and compared wave profiles to numerical simulations in order to evaluate the geometry concept as a research method in the field of energetic materials.

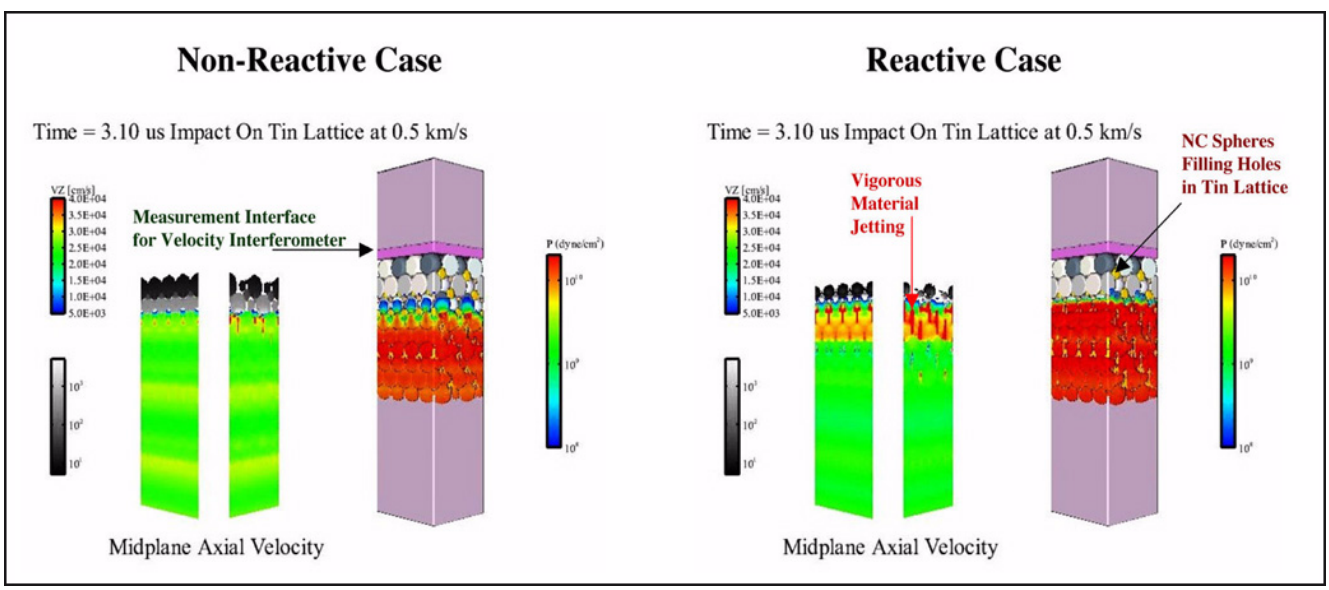
## Accomplishments

The first deterministic sample geometry considered was the concept of incorporating spheres of explosive material in the hole locations, in a close-packed bed of inert spheres. We compared wave propagation induced by sustained impact (such as that obtained in gas-gun studies): (1) in a sample bed of inert tin and non-reactive nitrocellulose spheres, and (2) in a sample containing reactive nitrocellulose spheres positioned in the tetragonal and trigonal hole locations in the tin sphere assembly. Dramatic wave amplification and induced material jetting are evident in the sample containing the energetic material. Such effects, at an interferometer window interface with the sample, should be accessible to the high speed and spatially resolved material motion sensing capability of a line-imaging velocity interferometer. Comparison of experimental data on these highly organized structures with numerical simulations could motivate the development of realistic models of "hot spot" initiation, growth, and coalescence.

The target design may incorporate several interesting variations (e.g., nitrocellulose replacing the tin spheres or located in only one class of hole location) that may aid in the interpretation of the relevant chemistry. While this design concept remains attractive, we were unable to procure nitrocellulose spheres in the appropriate sizes or in sufficient quantity and purity to build up the necessary gas-gun targets for experimental investigation. Experiments were performed on a surrogate design that utilized sensitized nitromethane (90% nitromethane/10% diethylenetriamine) as liquid explosive "filler" in a packed bed of tin spheres. Reaction growth was not observed with this explosive material in this design.

A second, very promising design involved nitromethane infiltration of a sintered Robocast alumina matrix with a well-defined periodic porosity. Samples incorporating nitromethane and sensitized

nitromethane (95% nitromethane/5% diethylenetriamine) were shock-loaded at impact velocities ranging from 0.79–1.2 km/sec. The wave profiles evaluated with both single-point and line-imaging velocity interferometers revealed globally dispersive but strongly organized (locally) stress states generated by shock propagation through the well-defined matrix. Vigorous explosive reaction was observed with the sensitized nitromethane and was likely present in the unsensitized nitromethane samples as well. We obtained representative VISAR traces of waves transmitted by the sensitized nitromethane/Robocast configuration. The reactive wave profiles strongly resemble those seen in heterogeneous explosive initiation with superimposed fluctuations indicating organized structures arising from "hot spot" like initiation and growth. Generating this type of reactive chemistry, in a clearly defined deterministic geometry, provides a unique window for investigation of the underlying hydrodynamics coupled to fundamental reaction mechanisms. For future studies, the characteristic length scales and periodicity of the open volume occupied by explosive material can be readily tailored by variation of the Robocast insert design. The methods and experimental results obtained in this one year project provide an important foundation for ongoing numerical simulations of deterministic geometry concepts.




*Wave propagation with inert* (left) *and reactive* (right) *nitrocellulose spheres positioned in hole locations of tin sphere lattice.*

# Micro Biomechanics

*79770*

*H. Sumali, M. Banning, A. D. Corwin, P. L. Reu*

## Abstract

The mechanical properties of the cell are of rapidly growing importance to the development of devices such as surface acoustic wave sensors and resonant micro-cantilevers for detecting bacteria. To advance the understanding of the mechanics of the cell, the objective of this project was to study several new methods to obtain mechanical and rheological properties of single biological cells. The project investigated the following:

(1) The use of an atomic force microscope (AFM) probe modified to apply a novel squeezing-flow technique to microsystems.

(2) The use of laser Doppler vibrometry (LDV) to measure the natural vibrations of certain cells.

(3) The development of a novel microelectromechanical system (MEMS) for obtaining high resolution force-displacement curves.

The squeezing-flow AFM technique utilizes two parallel plates, one stationary and the other attached to an AFM probe and driven with precise oscillations. Instead of using static force-displacement curves, the technique takes advantage of frequency response functions from force to displacement. Therefore, the technique obtains dynamic properties.

The LDV technique was used to investigate the finding, reported by others, that the membranes of certain biological cells vibrate naturally and that the vibration can be detected clearly with recent instrumentation.

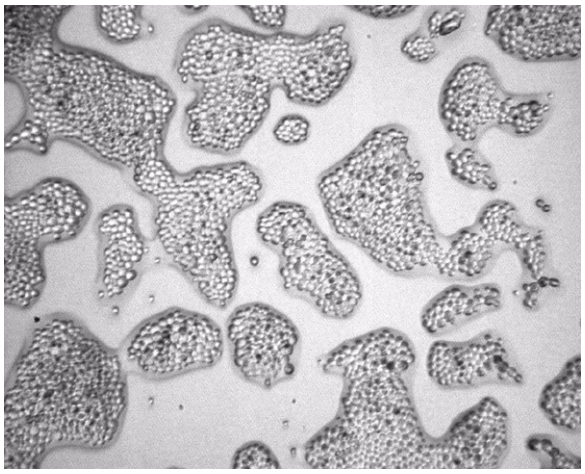
The MEMS device investigated in the third technique is a polysilicon surface-micromachined force sensor that is able to measure forces as small as a few piconewtons in both air and water. The simple device consists of compliant springs with force constants as low as 1.6 millinewtons/meter and Moiré patterns for nanometer scale optical displacement measurement.

## Accomplishments

(1) The use of an AFM probe modified to apply a novel squeezing-flow technique to microsystems.

Accomplishments include the following:

(a) Mechanical and rheological model of the human red blood cell.



*Colonies of* Saccharomyces cerevisiae *under a special microscope fitted with laser.*

(b) Design, fabrication, and testing of a special attachment to the AFM probe.

(c) A novel test method to measure mechanical and rheological properties of the human red blood cell.

(d) A theory relating the measurement results to the mechanical properties of the cell.

(2) The use of LDV to measure the natural vibrations of certain cells. The LDV technique proposed in this project appears to detect vibrations of certain cells. However, trials on *Saccharomyces cerevisiae* disproved the hypothesis that laser Doppler effects resulted in measurement of the cell membrane.

Accomplishments include the following:

(a) A new technique to measure vibration of single biological cells.

(b) Study on the sensitivity of the method.

(c) Limits to the applicability of the method. For example, trials on Saccharomyces showed that the vibrations of the cell membranes, reported in a publication by others, cannot be detected by laser-Doppler effects.

(3) The development of a novel MEMS for obtaining high resolution force-displacement curves.

We demonstrated that the force sensor was fully functional when immersed in aqueous buffer and when performing the kinesin inverted motility assay on the

sensor surfaces. These results show the force sensors can be useful for calibrating magnetic forces on magnetic beads and also for direct measurement of biophysical forces on-chip.

# Hydrodynamic Manipulation of Coalescence Dynamics

*79771*

*A. M. Grillet, C. J. Bourdon, A. D. Gorby, M. P. de Boer, C. F. Brooks*

## Abstract

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 opening 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. There is also currently no theory that 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.

## Accomplishments

Experimental work is progressing on two fronts: (1) the development of a novel flow control apparatus and (2) implementation of experimental methods to study coalescence. On the second point, we completed construction of an interferometry system that will allow the measurement of the thin film dynamics. The system was applied to study the dynamics of a captive drop approaching a solid surface in a quiescent medium. That work proceeded in parallel to the development of a flow system that allows the investigation of hydrodynamics on coalescence phenomena. Last fall, an initial design was developed that was simple and yet flexible for our needs. As part of the design process, we performed finite element simulations of the design concept to determine its effectiveness at generating a uniform extensional flow. Once the flow cell was constructed, we performed experimental measurements to determine the base flow pattern inside the cell, using particle imaging velocimetry. The flow in the center of the chamber is very axisymmetric and should provide a field of almost ideal flow for comparison to theoretical predictions of coalescence dynamics. In addition, we demonstrated the ability to manipulate the directionality of the flow using a computer controlled valve system. In short, we have surpassed all of our deliverables for the year to date.

We also made significant progress in development of the collaboration between Sandia and Professor Michael Loewenberg of Yale University. In addition to continued contacts through scientific conferences, in depth technical discussions were held during a visit to Yale in February. A contract is also in place to fund continued development of the coalescence theory that Professor Loewenberg has developed and to provide validation data for the ongoing experiments at Sandia.

# Control of Zero-Energy Modes and Contact for Nonlinear Solid Mechanics

*79772*

*W. M. Scherzinger, M. W. Heinstein, A. S. Gullerud*

## Abstract

Hourglass modes and contact in solid mechanics finite element applications are difficult to control. The hourglass control currently in our codes is based on small strain linear elasticity even though our codes are finite deformation codes. So while we have hourglass control, we still have problems with zero-energy modes. Contact algorithms on faceted geometries have focused on defining a set of node-on-face contact constraints. This construction inherently has dynamic perturbations due to surface normal discontinuities. Worse, yet, is that these perturbations often interact with hourglass modes and eventually can destroy a solution.

This work is an attempt to address these issues by first quantifying the problems and then correcting them. The research performed on zero-energy modes in FY 2005 was useful to the engineering sciences community. To begin, the theory behind the control of zero-energy modes, in particular the hourglass control algorithms in our solid mechanics codes, was re-examined and corrected. Many of the presentations that appear in the literature lack the mathematical rigor. Our research developed a new mathematical framework. After a new framework for the control of zero-energy modes was developed, hourglass measures were found. Much of the new mathematical framework went into the development of these measures. Finally, the measures were applied to a study of beam bending problems that examined numerical and analytical solutions. Mesh convergence studies were employed that show surprising results for coarse mesh accuracy when element aspect ratios are considered. In particular, the choice of hourglass parameters that affect course mesh accuracy is dependent on the element aspect ratio for these problems. This result is worrisome for more complex analyses with varying element aspect ratios.

## Accomplishments

The research this year concentrated on the control of zero-energy modes in our solid mechanics finite element codes. We accomplished a number of things to solidify our understanding of the underlying theory behind these modes and their control.

We began with the basic construction of the eight-node mean quadrature hexahedron. The published work (on which our elements are based) was slightly flawed. By fixing this flaw, we have a much more solid understanding of the element and what it is and is not capable of.

With this increased understanding, a number of techniques were developed to measure “hourglass” deformation in our elements. These methods are element-by-element as opposed to global measures of the deformation. Although we sought a global measure, we were unable to successfully develop such measure. We identified this as a future research idea.

We applied the hourglass measures to beam bending problems. The hourglass measures behaved as expected for this problem. However, other problems were seen regarding “coarse mesh accuracy.” In particular, we demonstrated that there is no single hourglass stiffness parameter that gives coarse mesh accuracy. This concept is, in the end, problem dependent. Factors that affect it are the particular boundary value problem, the element aspect ratios, and the material properties, among other things. The only reliable way to find an accurate solution is to use mesh refinement. A result of these last findings is that adaptivity may be the only reliable way to produce accurate solutions with the mean quadrature elements.

In addition to the work on our mean quadrature elements, we also examined a number of alternative element formulations (incompatible modes, enhanced strain, etc.) and worked on a new element formulation: the nodal based hexahedron. This is an element based on the nodal based tetrahedron developed at Sandia. These pursuits showed promise and may develop into other research topics.

# Atomic Scale Modeling of Phonon-Mediated Thermal Transport in Microsystems

*79773*

*C. J. Kimmer, S. Aubry, E. S. Piekos, E. B. Webb III, L. M. Phinney*

## Abstract

Thermal management is increasing in importance as the national security mission at Sandia drives the development of micro- and 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 sub-grid physics input to microsystem device design modeling, generating a robust multiscale description of thermal transport. Pre-existing and novel molecular dynamics (MD) techniques will be used to perform simulations, explicitly examining phonon-microstructure and phonon-phonon interactions across a range of temperatures and providing a detailed characterization of phonon transport at the atomic scale. The simulation results will be analyzed to produce sub-grid models of phonon transport for use in Sandia's mesoscale models that, 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.

## Accomplishments

The focus of the first year of this project was on developing the MD methods needed for the atomic scale modeling of phonons interacting with grain boundaries. To this end, we developed a suite of computational tools to generate initial conditions corresponding to phonon wavepackets of arbitrary wave-vector and polarization in silicon for use in MD simulations. We reproduced published results to verify our MD code and the phonon initial conditions. Additionally, we characterized the suitability of atomic scale methods to capture thermal effects on phonon dispersion (i.e., frequency shifts) and lattice thermal expansion. Although these effects have been shown to be negligible for an ideal, ordered interface, some small differences due to thermoelasticity are observed for a twist grain boundary. Nonetheless, the thermoelastic differences are smaller than the differences arising as a function of twist angle for twist grain boundaries. Consequently, the dominant thermal effects on phonon scattering will be disorder at the interface and the orientation mismatch between grains. We also developed an MD method for simulating phonons that are non-normally incident on the grain boundary plane, allowing for the complete modeling of the parameter space needed to describe mesoscale models. Currently, we completely simulated the scattering of all possible acoustic phonons off the $\Sigma$ 29 (001) twist grain boundary in silicon. These data are generating our first frequency dependent mesoscale model for grain boundary scattering. This complete modeling of a grain boundary also used our novel capability to simulate and analyze many wave packets simultaneously, as opposed to characterizing one wave packet per simulation. This capability decreased the computer time required to characterize a given grain boundary by roughly an order of magnitude.

We are transferring data generated by the MD simulations to the mesoscale direct simulation Monte Carlo (DSMC) code to produce an initial grain boundary scattering model. This ability to model frequency dependent grain-boundary scattering was recently added to the DSMC code for this project. For

experimental validation of the DSMC models, we are conducting thermal conductivity measurements for Sandia Ultraplanar Multilevel MEMS Technology V (SUMMiT V™) polysilicon layers using a steady-state test method. We completed the first set of measurements, and future testing will focus on reducing the uncertainty in the measurements by testing more samples and using the three-omega technique to provide a second method of thermal conductivity determination.

## Other Communications

C.J. Kimmer and E.B. Webb III, "Phonon Scattering By Molecular Dynamics: Temperature Dependence and Effect of Structural Disorder," presented at the American Physical Society March Meeting, Los Angeles, CA, March 2005.

# Multiphase Dynamics of Soft Biological Tissues

*79774*

*R. E. Jones, T. D. Nguyen, B. L. Boyce, M. Okandan*

## Abstract

We are applying expertise at Sandia in micromechanical experimentation and continuum mechanics modeling to soft materials of biological origin. Biological tissues are unique structural materials with technologically appealing properties. Soft tissues, such as skin, are constructed from a composite of solid and fluid components. This is the first coordinated experimental/modeling project at Sandia to consider the mechanics of fibril-based anisotropy, viscoelasticity, and the coupled solid-fluid deformation of soft biological tissues. The project will provide detailed understanding of the deformation of ocular tissues, specifically the highly structured skin-like tissue in the cornea. This work will improve our knowledge of soft materials testing and modeling. It will also improve our knowledge for designing new biomedical microelectromechanical systems (MEMS) prosthetics and for understanding collateral damage caused by munitions. Furthermore, clear understanding of the mechanics of soft tissues will lead to bio-inspired materials or applications of natural materials, such as highly supple and impact resistant body armor. The models developed in the project may also be applicable to modeling cell deformation for disease identification.

## Accomplishments

In the first year, we chose to study corneal tissue, the transparent layer in front of the lens, as our model tissue because it is a simplified and very structured type of skin. It is avascular and lacks many of the non-structural components of ordinary skin (as would a synthetic analog). It also has fibrils arranged in regular lamellae, unlike the random cross-linked arrangement in ordinary skin.

Issues we are attempting to address in our experiments and modeling are: (1) anisotropy due to fibril arrangement into structured lamellae needs to be incorporated, (2) the assumption that matrix is the primary source of viscosity in solid needs to be tested, (3) viscosity of matrix needs to be distinguished from viscosity due to fluid flow via experiments, and (4) intake of free water (swelling) and its effect on mechanical response needs to be quantified and understood.

We conducted preliminary unconfined compression testing and simulation of the lens. Displacement and load controlled tension tests on corneal strips cut along nasal-temporal (N-T) and inferior-superior (I-S) directions were conducted to determine material response and viscoelastic model parameters in the two primary directions. In addition to working out many details of specimen preparation and fixturing, we now have a regimen that gives us reliable, repeatable data. We are using left-right pairs from the same organism to winnow out individual variations.

With these data, our viscoelastic model of the cornea is progressing, and we are discovering new qualitative features in the data that reveal physical mechanisms and microstructure. The nonlinear finite-deformation Reese-Govindjee based model shows fidelity with the observed behavior in fitting and prediction, with the only significant exception being strain recovery at low driving stresses.

To model the anisotropy of the cornea, the first step was to fit an isotropic viscoelastic model to N-T and I-S data independently to determine the relative material response in the two primary directions. This isotropic model is intended to solely represent the fibril network in a particular direction, neglecting the response of the proteoglycan for now.

To account for the multiphase nature of soft tissues, we are developing a dynamic biphasic mixture theory for modeling coupled solid deformation and fluid flow through porous tissue. We assume the solid includes collagen fibrils and proteoglycans, while the fluid is the interstitial fluid (mostly water) that is “free” to flow through the porous solid matrix.

# Capabilities for Design and Analysis of Fluid Structure Interaction for Active Aeroelastic Control

*84438*

*J. L. Payne, J. D. Hales, C. C. Ober, S. J. Beresh, M. F. Barone, J. E. Massad*

## Abstract

Computer simulation of the interaction of flexible structures with fluid flow is a burgeoning research field. The present effort is aimed at answering two questions: (1) What simulation technologies for fluid/structure interaction are available, and how are they best assembled to enable accurate and efficient calculations? and (2) How can reduced order modeling techniques be applied to data generated by such simulations to enable efficient application in a design and analysis setting? The first question is addressed by a literature survey of the state of the art in computational aeroelasticity. Numerical schemes for coupling fluid and structural dynamics solvers are reviewed and characterized according to their accuracy, stability, robustness, and efficiency. The second question is explored by investigation of the proper orthogonal decomposition (POD)/Galerkin projection approach to reduced order modeling of a coupled fluid/structure problem. In this approach, the high-fidelity coupled simulation data are used to construct a reduced order model that is able to mimic the behavior of the full simulation but at a much lower computational cost. The mathematical formulation for such an approach that can be applied to problems with a moving fluid mesh and/or time-dependent boundary conditions is presented.

## Accomplishments

We conducted a literature survey of state of the art numerical techniques for computational aeroelasticity. We organized the findings into separate sections of a report addressing: fluid mesh motion, geometric conservation law, time advancement schemes, wetted surface interface schemes, and selected applications. Certain common themes were identified, the most important of which is the numerical conservation of energy between the fluid and structure systems. Examples from applications indicate that computational analysis of complex fluid/structure systems is still an expensive proposition.

The focus of the project then shifted to exploration of ideas for increasing the efficiency of a fluid/ structure analysis using reduced order modeling. Reduced order models (ROMs) are derived from simulation data sets over some limited input parameter range. Solutions to ROMs are much cheaper to compute than the full simulations because the number of retained degrees of freedom is much lower. The POD/Galerkin approach for coupled fluid structure problems was examined in detail. We derived the mathematical foundations for POD/Galerkin ROMs of coupled fluid/structure systems. Special considerations for this class of problems include accommodation of a moving fluid mesh and unsteady boundary conditions for the fluid system. We developed and documented strategies for handling these two issues. We proposed two methods for building a coupled ROM from separate fluid and structural ROMs. The first assumes that the structural displacements are small, so that the fluid system is defined and solved on a static mesh. The second makes no such assumptions and involves mesh motion as part of the ROM. The second strategy, while more general, promises to be more complex and costly. We also proposed test problems for studying these methods of building fluid/structure ROMs as future work. Finally, we identified potential sources of error in ROMs as a precursor to the development of a verification and validation strategy for ROMs. These include basis truncation, sampling error, numerical quadrature error, and temporal integration error.

## Other Communications

M.F. Barone and J.L. Payne, “Methods for Simulation-based Analysis of Fluid-Structure Interaction,” Sandia Report SAND 2005-6573, Albuquerque, NM, 2005.

# Materials Science and Technology

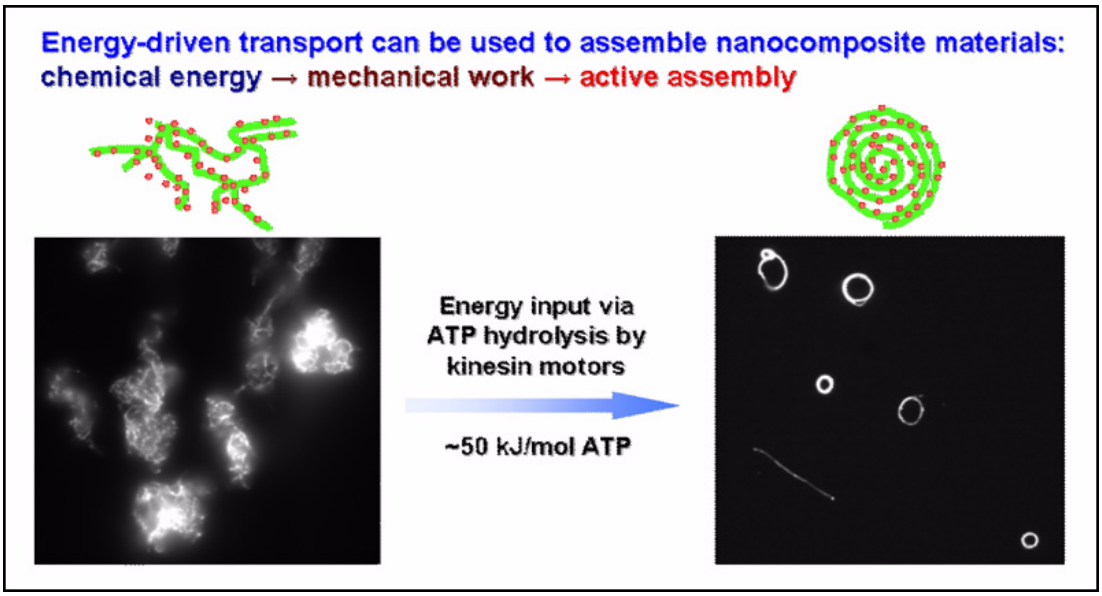
## Assembly and Actuation of Nanomaterials Using Active Biomolecules

*52531*

*G. D. Bachand, A. Carroll-Portillo, A. M. Trent, M. Bachand, M. Farrow, E. D. Spoerke, C. M. Matzke, B. C. Bunker, S. J. Hearne, M. P. de Boer, R. P. Manginell*

### Abstract

The formation and function of living materials are fundamentally different from those of synthetic materials and devices. While synthetic materials tend to have static structures and are not capable of adapting to changing environmental conditions, living systems use energy to create, heal, reconfigure, and dismantle materials in a dynamic, non-equilibrium fashion. The overall goal of this research project is to organize and reconfigure functional assemblies of nanoparticles using strategies that mimic those found in living systems. In the first strategy, active assembly of nanostructures, we explored using principles, similar to those used by diatomaceous algae to assemble their silica shells, to actively assemble nanocomposite materials using microtubule filaments and synthetic nanoparticles. In FY 2005, we established "design rules" that enable the transport of a wide array of nanoparticles cargo using kinesin and microtubules. We also characterized interactions among nanoparticle cargo. In the case of large cargo (~1 micron), we observed a range of events, including transfer of particles between microtubules. Smaller cargo such as nanocrystal quantum dots (nQDs) interacted differently, and we used the process to actively assemble nanocomposite structures.




*Active assembly of nanocomposite springs.*

In the second strategy, responsive reconfiguration of nanostructures, our goal was to exploit kinesin and microtubules to mediate the optical properties of nQDs by modulating the spacing among particles, mimicking the strategy used by fish to change the color of their skin. We developed two methods for assembling three-dimensional microtubule structures, mimicking those found in the melanophore cells of fish. Our preliminary attempts to control dynamic energy transfer using kinesin-based transport of nQDs on these arrays were unsuccessful. Lastly, we used microcantilever-based microelectromechanical systems (MEMS) devices to calibrate a magnetic force tweezer instrument, which we used to measure the collective force output by surface-tethered kinesin. Overall, we were able to successfully demonstrate the active assembly of nanostructures and realize several key steps in developing responsive nanostructures.

## Accomplishments

Our FY 2005 milestones were aimed at: (1) standardizing the conditions necessary for nanoscale cargo transport, (2) characterizing interactions between microtubules carrying nanocargo, (3) demonstrating the active assembly of nanocomposite materials, (4) developing three-dimensional assemblies of microtubules fibers, (5) demonstrating dynamic energy transfers based on kinesin transport of nanoparticles, and (6) exploiting MEMS sensors to characterize motor protein mechanical forces. Overall, we achieved excellent progress.

Previously in this project, we demonstrated the ability to transport metallic and semiconductor nanoparticles using kinesin and microtubules. This year our goals were to understand the critical conditions and to define a set of “design rules” for supporting active nanoparticle transport. In our experiments, we determined that nanoparticle size and density on microtubule shuttles define the kinesin based transport characteristics. From these rules, we established a standardized method for transporting a range of nanoparticle cargo.

A key objective was to understand and control the loading and unloading of cargo from microtubule shuttles. With regard to large (e.g., 1 micron) cargoes, interaction among particle carrying cargo resulted in six distinct events: (1) no cargo transfer, (2) cargo transfer, (3) microtubule breaking, (4) forcible release of the cargo, (5) bending of microtubule with no cargo transfer, and (6) microtubule aggregation. Temperature significantly affected the distribution of these events, and it is likely these effects are due to changes in the bond strength as a function of temperature, as well as the loading rate during a collision.

We observed that interaction among microtubules carrying relatively small cargo (e.g., nQDs) leads to a fundamentally different outcome: active assembly of nanocomposite materials. The intrinsic multivalency of streptavidin, which is used to link nanoparticles to biotinylated microtubules, serves as “molecular glue” whereby multiple microtubules can bond to one another and assemble into structures. Random aggregation of quantum dot coated microtubules forms large, unorganized structures in the absence of kinesin; whereas, kinesin based transport of quantum dot coated microtubules leads to the assembly of ordered, circular composites. We achieved a significant proof of principle demonstration of the active assembly of these nanocomposite materials using energy driven processes (i.e., kinesin transport). Further, these assembled composites are capable of storing a considerable amount of energy (> 500,000 kT at room temperature).

An overarching goal of this project was to demonstrate dynamic energy transfer in a manner analogous to how fish change color. To this end, we developed two strategies for assembling three-dimensional assemblies of microtubules that are directly analogous to those found in the melanophore cells of fish. One of these strategies provides polar orientation of the microtubule filaments that can enable programmed, bidirectional transport of nanoparticles. Subsequently, we placed significant effort on demonstrating dynamic energy transfer via kinesin transport using these assemblies. Although we successfully attached kinesin to nanoparticles, we were unable to accomplish transport on the three-dimensional assemblies.

In FY 2004, we fabricated microcantilever beams for measuring the mechanical forces produced by motor proteins using the Sandia Ultra-planar Multilevel MEMS Technology 5$^{TM}$ (SUMMiT V$^{TM}$) process. This year, we used these MEMS sensors to calibrate a new “magnetic force tweezer” instrument that was assembled in a separate project. We then used the magnetic force tweezer to measure the collective force exerted by surface tethered kinesin during the transport of microtubule shuttles. A magnetic sphere (0.5 micron) was linked by biotin-streptavidin to the end of a microtubule. We then used surface tethered kinesin to transport the microtubule/sphere complexes. By in-

creasing the magnetic force, we used the tweezer to determine the "stall force" for the collection of motors.

In summary, we achieved critical milestones and developed a number of key technologies. We achieved one of the primary goals of the project (demonstrating active assembly of nanomaterials) and realized significant advances with respect to the second goal (demonstrating dynamic energy transfer).

## Refereed

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## Other Communications

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G.D. Bachand, S.B. Rivera, A.K. Boal, M. Bachand, J. Liu, and B.C. Bunker, "Active Biological Transport Systems as Functional Components of Nanoscale Materials and Devices," presented at the Materials Research Society Spring 2005 Meeting, San Francisco, CA, March 2005.

M. Bachand, A. Carroll-Portillo, A. Trent, M. Farrow, E.D. Spoerke, J. Liu, B.C. Bunker, S. Jeong, M. Achermann, V. Klimov, J. Hollingsworth, and G.D. Bachand, "Kinesin and Microtubule-Based Transport Systems: Nanoscale Science & Engineering Perspectives and Applications," presented at the NIBIB-DOE Nanobiotechnology Workshop, Bethesda, MD, March 2005.

E. Spoerke, A.K. Boal, G.D. Bachand, B.C. Bunker, J.A. Voigt, and J. Liu, "Directed Assembly of Microtubule-Based Architectures," presented at the Materials Research Society Spring 2005 Meeting, San Francisco, CA, March 2005.

A. Trent, A.K. Boal, B.C. Bunker, and G.D. Bachand, "Secondary Control of Active Biological Transport Systems for Dynamic Nanomaterials Synthesis," presented at the Materials Research Society Spring 2005 Meeting, San Francisco, CA, March 2005.

# Decomposition of Contaminants Using Photochemically Active Nanoparticles

*52698*

*K. F. McCarty, B. A. Simmons, T. Zifer, R. A. Kemp, T. J. Boyle, D. R. Jennison, D. H. Morse, E. E. Tarver III*

## Abstract

The decomposition of hazardous biological (viruses and bacteria) and nonbiological (e.g., nerve agents, $H_2S$, HCN) compounds can be realized using a new class of photochemically active nanoparticles that have been fine tuned for decontamination applications. Several metal oxides ($TiO_2$, ZnO, $Fe_2O_3$, $WO_3$) have demonstrated the ability to destroy contaminants upon exposure to ultraviolet (UV) light. However, to make them practical to use, improvements in their activity are necessary. These materials work by photoactivating water and oxygen to create highly reactive species (e.g., OH•) that readily decompose the compounds described above. We investigated a new class of highly photoactive materials combining scientific inquiry into the mechanisms of photocatalysis, advanced techniques to synthesize nanoparticles, and testing to determine the ability of these materials to destroy simulants. We used first-principles calculations to establish how $TiO_2$ can be doped to absorb visible light. Using in situ microscopy, we established that the photocatalytic activity occurred uniformly over a $TiO_2$ surface. We synthesized and evaluated a large number of nanomaterials, exploring two main nanoparticle synthesis routes: (A) solution routes and (B) solvothermal routes. We designed and constructed specialized test equipment. We discovered a large number of doped $TiO_2$ materials that were less active than undoped $TiO_2$. However, our testing showed that several materials we synthesized were more catalytically active than any undoped material, including newly commercialized $TiO_2$ nanoparticles.

## Accomplishments

We developed several new methods to synthesize doped $TiO_2$. We found two approaches to be most useful: coating $TiO_2$ nanoparticles with metals (a "core shell"), and coating particles with $TiO_2$ (a "shell core"). Using the former approach, we successfully deposited fine lanthanum metal particles on $TiO_2$ nanoparticles. Using the latter approach, we synthesized a $TiO_2$ shell on silver nanoparticles. Most importantly, photocatalytic testing established that the lanthanide doped materials exhibited activity with visible light. While synthesizing metal lanthanum nanoparticles proved to be difficult, we did successfully implement a procedure to make nanoparticles of lanthanide oxides.

We also developed a powerful capability to test materials for photocatalytic activity. We completed the assembly and testing of a powerful reactor. Our computer controlled system enables the simultaneous evaluation of eight materials under exquisitely controlled and characterized conditions. We can test activity in both the UV and visible light regimes. Our second major achievement was developing the ability to test the biocidal action of the photocatalysts to kill cells. We developed a procedure to quantify the biocidal activity of photocatalysts to deactivate cells of *Escherichia coli*. Our initial tests established that $TiO_2$ nanoparticles readily killed *E. coli* cells.

Our materials development was aided by our efforts to understand the underlying physical mechanisms using first-principles electronic structure calculations and in situ microscopy. We studied how nitrogen doping causes $TiO_2$ to adsorb light in the visible spectrum. Based on our calculations, we submitted a detailed picture for publication. Finally, we successfully imaged the process of photocatalysis occurring on a surface. We used low energy electron microscopy to image the adsorption and photocatalytic oxidation of trimethlyacetic acid (TMA) on the $TiO_2$ (110) surface. We observed that both the adsorption and decomposition of the TMA, which caused large contrast changes, occurred uniformly across the surface. Atomic steps on the substrate appeared to play no special role.

## Refereed

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S.D. Daniel, T.J. Boyle, K.F. McCarty, B.A. Simmons, R.A. Kemp, and T. Zifer, "Decomposition of Contaminants Using Photochemically Active Nanoparticles," presented at the 229th National Meeting of the American Chemical Society, San Diego, CA, March 2005.

T.J. Boyle, L.M. Ottley, L. Tribby, S.D. Daniel, S.D. Bunge, B.A. Simmons, P.G. Clem, and C.J. Brinker, "Lanthanide Alkoxides for Production of Ceramic Materials," presented at the American Chemical Society Annual Meeting, San Diego, CA, March 2005.

K.F. McCarty, "Low-Energy Electron Microscopy as an In Situ Diagnostic for Catalysis," presented at the Workshop on Opportunities in Nanocatalysis, Tarrytown, NY, October 2005.

# Thermally Cleavable Surfactants

*52699*

*B. A. Simmons, C. S. Yun, T. M. Long, T. Zifer, K. Rahimian, G. M. Jamison, J. R. McElhanon*

## Abstract

Surfactants are molecules that have the ability to self-assemble into a variety of supramolecular structures. These structures, and the intrinsic amphiphilic nature of the oil-water interface they possess, have been used in numerous laboratory and industrial processes, including the synthesis of advanced materials. A major problem associated with the use of surfactants is their subsequent removal after use and the recovery of segregated materials. We successfully developed three novel approaches to surfactant removal using (1) thermally degradable surfactants, (2) metathesis depolymerization that contain weaklinks internal to the molecule itself, and (3) cleavable surfactants based on nitroso chemistry. The thermal degradation approach is based upon a reversible Diels-Alder reaction that has already found utility at Sandia in the preparation of removable encapsulants, foams, adhesives, and dendrimers. The metathesis depolymerization degradation approach is based upon a catalyzed alkane ring forming reaction of the surfactant that has been widely used in polymer science.

Surfactant removal becomes very significant in the realm of extended mesoporous nanosized structures, such as ceramics, polymers, inorganic nanocrystals, and polymer-ceramic composites. The present technique of material recovery is typically a combination of centrifugation, calcination, and solvent washing that destroys the desired architecture and functionality of the synthesized material. We believe that the incorporation of a cleavable linkage would solve this problem by allowing for the removal of the surfactant molecules through the formation of a hydrophilic and hydrophobic section. We successfully synthesized a wide range of cleavable surfactants that will function as removable templates.

## Accomplishments

Our research is centered on the incorporation of cleavable elements into surfactant molecules that, when activated, will degrade the surfactants into hydrophilic and hydrophobic elements. This will effectively remove the surfactant from the system entirely and eliminate the surface activity of the system. We met and surpassed all of the project milestones.

We successfully developed three unique paths to create cleavable surface active materials. The first involves the use of a catalyzed metathesis depolymerization element within a surfactant molecule that forms alkene rings from the hydrophobic tail section of the surfactant when activated. The second approach utilizes nitroso based weaklinks within surfactant molecules. The third technique involves the use of thermally cleavable Diels-Alder linkages located within various types of surfactant molecules and assemblies. We synthesized both monomeric and linked-dimeric (Gemini) surfactants, and we are in the process of fully characterizing them.

We characterized these surfactants by surface tensiometry, nuclear magnetic resonance (NMR), and small angle neutron scattering (SANS). These surfactants are unique and have not been reported in the scientific literature. We attempted to synthesize a third class of labile surfactants based on nitrosochloride chemistry. We demonstrated that these surfactants can be used to synthesize polymeric and silicate materials. There is significant interest from the biotechnology community in using these materials for enhanced sample preparation that precedes mass spectrometry analysis. In addition, the Diels-Alder based materials have generated industrial interest.

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# Transition Metal Catalyzation of Complex Hydride Absorption/Desorption Reactions

*52700*
*E. H. Majzoub, R. R. Stumpf*

## Abstract

Sodium aluminum hydride represents an advance in hydrogen storage technology. This complex ionic hydride holds over 7 weight percent hydrogen, and when doped with Ti, it reversibly releases about 4.5 weight percent hydrogen. While this is an advance over the traditional interstitial hydrides (at less than 2 weight percent hydrogen), there are materials properties of the interstitial compounds that are highly advantageous. For example, it is desirable to alloy the interstitial compounds, such as $LaNi_5$, with other metals in order to tune the thermodynamic stability of the hydride phase, which determines the plateau pressure of the compound. The plateau pressure is very important for practical applications and will determine the operating temperature of the hydride. Therefore, we attempted to form mixed alkali compounds and accomplished the formation of $K_2LiAlH_6$.

We addressed the mechanism of the enhanced kinetics through experiments using Pd as a catalyst. Sorption experiments with Pd powder and Pd foil indicate that formation of $NaAlH_4$ is possible without the transition metal catalysts; however, the sorption rates are much slower.

We addressed compound formation via solution or solid doping of $NaAlH_4$ with Ti. The Ti containing phases are very different; this will impact efforts to manufacture large quantities of this compound for practical purposes.

## Accomplishments

Among our original milestones for FY 2005 was the demonstration of hydrogen sorption in mixed $Na_{(1-x)}Li_xAl_{(1-y)}B_yH_4$. We modified this goal after it became apparent, in the literature and in our own experiments, that these compounds do not support continuously variable substitution but appear to be strict line compounds. The compound $K_2LiAlH_6$, a lithium substituted potassium alanate, was recently described in the literature. The crystal structure of this compound puts the Li and K cations on strictly separate symmetry sites. There is no evidence of continuous composition variability. This result impacts the thermodynamic tunability of these compounds in comparison with interstitial compounds.

We performed sorption experiments using Pd foil and Pd powder as a hydrogen dissociation catalyst. The experiments showed that alanate formation occurs upon having NaH, Al, and atomic hydrogen in contact, without the presence of Ti or other transition metal "catalysts." The sorption kinetics of the reaction are very slow, however, and suggest that while dissociation of hydrogen is important, and allows the formation of $NaAlH_4$, it does not appear to account entirely for the dramatic increase in kinetics.

We performed multiple-quantum magic angle spinning nuclear magnetic resonance (MQ MAS NMR) experiments on solution Ti doped and solid Ti doped samples of $NaAlH_4$. We prepared the solution doped samples in tetrahydrofuran (THF); this resulted in very different compound formation compared to solid doping via mechanical milling. Mechanically milled samples show evidence of $TiAl_3$, a common intermetallic in the Ti-Al system, while solution doping results in no $TiAl_3$ formation. The results from our NMR experiments are consistent with the formation of nanoparticle Al-Ti that forms in conjunction with the strong interaction of Ti with THF molecules in the solvent. Solution doped samples could be made active by mechanical milling. The results of these experiments will impact the production methods for large quantities of $NaAlH_4$.

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# Quantification of Environments and Surfaces Within Micropackages

*52701*

*S. M. Thornberg, J. A. Ohlhausen, D. E. Peebles, D. R. Tallant, K. R. Zavadil, M. R. Keenan, R. A. Plass, M. S. Kent*

## Abstract

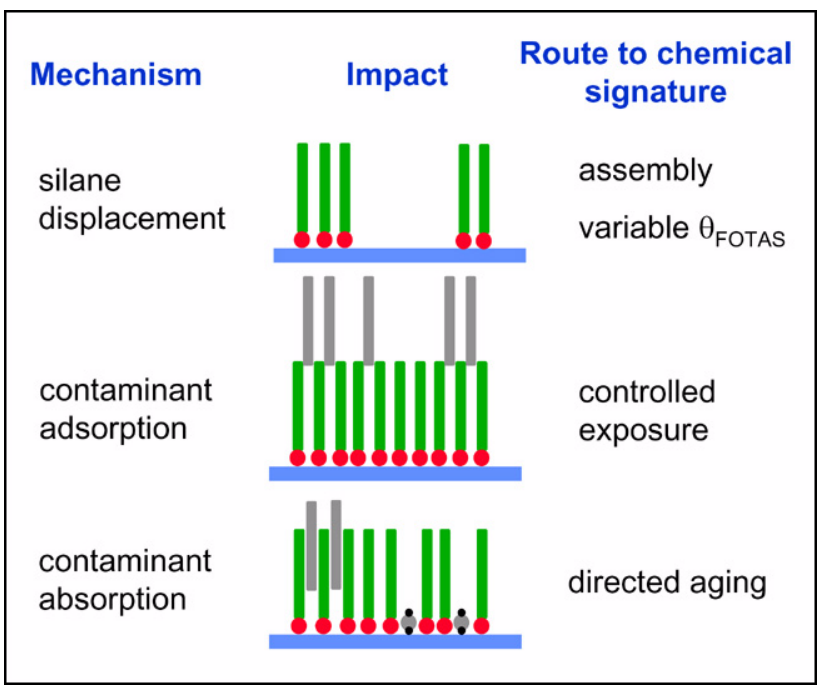
Chemical and physical materials aging processes can significantly degrade the long term performance and reliability of dormant microsystems. This degradation results from materials interactions with the evolving microenvironment by changing both bulk and interfacial properties (mechanical and fatigue strength, interfacial friction and stiction, electrical resistance). Eventually, device function is clearly threatened, and these aging processes have the potential for high negative consequence.

The goal of this project is to develop a set of tools that are analytical strategies and methods to measure the spatially resolved chemical inventory within Sandia fabricated and packaged microsystems. These tools can be broadly subdivided into surface and internal gas atmosphere characterization with the aid of multivariate data processing. Both require the design and fabrication of a suitable microelectromechanical system (MEMS) test platform that opens easily and cleanly. Tools developed in this work will enable the acquisition and processing of surface/gas data to support the design of integrated state-of-health sensors, to identify important degradation/dormancy mechanisms, to establish packaging specifications to assure adequate weapons reliability, and to establish future surveillance methods.

## Accomplishments

Molecular level films are a preferred way of tailoring surface properties in MEMSs. Sandia's approach to minimizing intersurface forces is by using self assembled monolayers (SAMs). Sandia's investment in FOTAS (1H,1H,2H,2H-perfluorooctyltris(dimethylamino)silane) as the preferred SAM for MEMS applications prompted us to focus on the FOTAS-Si system.

The first challenges of this project were to characterize and quantify the SAM film and learn how to quantify the state of the film. We chose specific degradative mechanisms and designed experiments to identify the analytical techniques needed to characterize the film. Finally, we developed and demonstrated a suite of analytical methods that we applied to packaged MEMS devices.




*Proposed mechanisms and schematic impact of SAM degradation on a Si substrate. Approaches for determining what signatures might accompany these mechanisms are also given.*

We developed time-of-flight secondary ion mass spectrometric (TOF-SIMS) methods to quantify the surface coverage of FOTAS and generate a unique chemical signature that indicated local SAM displacement. We developed sophisticated algorithms to extract useful information from these very large data sets. We performed multivariate curve resolution by alternating least squares (MCR-ALS) with the addition of "spatial simplicity" constraints, and we accommodated the energy shift by equality constraining two spectral components to equal the derivatives of their respective pure component spectra. These latter equality constraints enabled separation of the chemical information from the instrumental artifact.

We used x-ray induced photoelectron emission to generate the absolute measurement of surface coverage for specially prepared samples that were eventually used to calibrate the TOF-SIMS quantitation algorithms. Specifically, the quantity of interest is the number of FOTAS molecules per unit area or areal density. We demonstrated that monochromatic x-ray photoelectron spectroscopy is a quantitative tool for determining the SAM surface concentration on MEMS device level substrates. With this capability, surface properties can be monitored to track changes as the surface is modified during packaging, use, and aging.

Atomic force microscopy is one of the higher spatial resolution techniques available for surface characterization. Resolution is limited by several factors: the tip radius (with 1 nm tips commercially available), the magnitude of the tip/surface force field being detected, a combination of how the field propagates through space and how it is sensed (field or field gradient), and lastly by the surface topography. Our data show that rough polycrystalline surfaces (50 nm average height variation) can be probed using the scanning Kelvin force microscopy technique to look for subtle variations in the silane surface film produced by tribological wear.

Finally, we developed methods to determine the hermeticity of MEMS packages using gas analyses. The most definitive way to assess the hermeticity of the package is to sample and analyze the gas. MEMS packages are of various sizes and have internal volumes that range from about a milliliter down to nanoliters. Analysis of gases within milliliter sized volumes is challenging enough with conventional technology; nanoliter sized volumes are impossible. We developed methods that are radically different from standard techniques because of the custom hardware used and the pulsed method for gas introduction into the residual gas analyzer. This change enables not only the analysis of nanoliter sized MEMS packages, but also enables a rapid way to analyze the gases repetitively in a statistically significant manner (e.g., gas from each package was analyzed dozens of times during a 20 minute time period).

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# Assembly of Ordered Electro-Optical and Bioactive Materials and Composites

*52702*
*C. J. Brinker, B. A. Simmons, F. B. Van Swol*

## Abstract

The discovery of intrinsic metal-like conductivity in conjugated polymers represents a significant milestone in polymer chemistry and materials science and was recently recognized by the 2000 Nobel Prize Award in Chemistry. This project aimed to address several key issues related to the performance and utility of conductive polymers. Our objectives were to: (1) develop new methods to assemble ordered conducting materials on multiple length scales, (2) establish fundamental structure-property relationships of nanostructured conjugated polymers, and (3) integrate the new materials/structures with microelectronic/microfluidic devices.

We fabricated nanoporous electrodes with uniform pore sizes using surfactant self-assembly techniques and functionalized the nanoporous electrodes. When the target biomolecules are captured in the pore channels through specific binding or hybridization, the nanopores are totally or partially blocked. As a result, the access of indicator molecules to the conductive surface of the electrode is affected and the electrochemical response on the electrode is reduced. This mechanism is sensitive to the presence of nanomole ($10^{-9}$ mole) to femtomole ($10^{-15}$ mole) level molecules, and has potential as a simple, general, and labeling-free technique for electrochemical biomolecular recognition.

## Accomplishments

Sensitive and labeling-free electrochemical sensing has attracted wide attention for biomolecular detection. We achieved the synthesis and utilization of self-assembled nanoporous electrodes for such applications based on surfactant directed self-assembly. We functionalized the electrodes to interact with specific biomolecules. We studied the pore blocking behavior in the presence of proteins and deoxyribonucleic acid (DNA) molecules. Our results showed that these nanoporous electrodes have potential as a simple, general mechanism for labeling-free biomolecular recognition from nanomole to femtomole levels.

First, we coated a 0.1 micrometer thick hybrid nanoporous silica film containing amine functional groups over a conductive electrode using surfactant self-assembly and dip coating. The film has a continuous, disordered, and uniform pore structure (pore diameter is about 8 nm). In the next step, we attached biotin groups, or oligonucleotides (probe molecules), to the amine functional groups. Finally, we reacted the functionalized electrodes with streptavidin, or complementary (target) or noncomplementary (non-target) DNA molecules. We then measured the electrochemical response in an electrolyte solution containing indicator molecules.

We investigated a simple, but sensitive, pore-blocking method based on self-assembled nanoporous electrodes that can be potentially used for labeling-free biomolecular recognition. Compared to previous studies, this approach is more generalized and is not limited to particular types of molecules. We observed nanomole to femtomole level sensitivity. The materials and electrode fabrication, the sample preparation, and electrochemical testing procedures are simple, straightforward, and fast.

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# Advanced Packaging: Joining Technology for Microsystems

*52703*

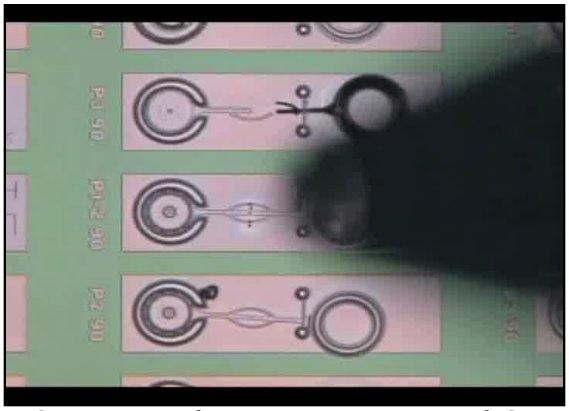
*G. A. Knorovsky, E. A. Holm, J. R. Michael, C. V. Robino, D. O. MacCallum*


## Abstract

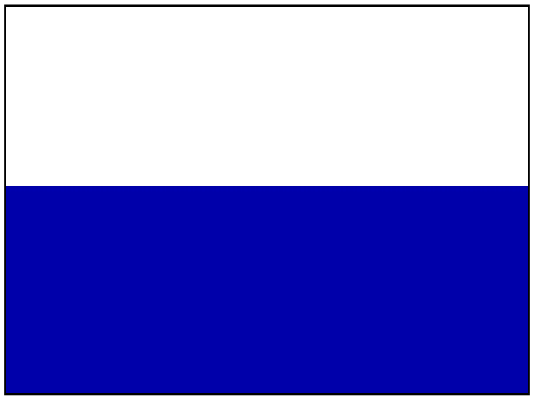
Current microelectronics packaging technologies are inadequate for potential microsystems applications. This project investigated two technologies that can address these key microsystem needs: micron scale high energy density (HED) fusion welding and focused-ion-beam deposition joining (FIBDJ). At the micron scale, materials properties that dominate joining response differ appreciably from those at macroscopic sizes. We characterized the materials properties/process interactions that control microscale joinability in HED welding and FIBDJ.

Lasers and electron beams (e-beams) are feasible processes for HED welding. Lasers are infeasible at < 10 microns because of focusing limitations. Previously, we used a slightly modified scanning electron microscope (SEM) to demonstrate e-beam microwelding; however, our modeling results showed that, although smaller than a laser, the target's electron transparency at SEM accelerating voltages (AVs) diffuses the e-beam to a few microns diameter and modifies its energy deposition in the target. Thus, penetration depth can be controlled by AV. For example, for a two layer microelectromechanical system (MEMS) lap weld, higher AV would be preferable to deposit energy below the top layer. Conversely, for a single layer butt weld, a lower AV would be preferable.

FIBDJ avoids the SEM's beam spreading, as the beam's impact is only at the surface, where it "cracks" an adsorbed organometallic gas. Further, the extremely low current FIB beam is smaller than that used in SEM HED welds. Thus, it may be applied at submicron scales. We measured mechanical properties (aided by a finite element analysis of the test specimen) of the ambient substrate temperature Pt organometallic deposit. We created a lattice gas cellular automata based model to establish flow effects around geometrical discontinuities for comparison with FIBDJ process characterization data.

*Video shows testing of MEMS FIBDJ specimen; failure occurs at locations predicted by the finite element method model.*



*Video shows GOMA simulation of recoil driven molten metal flow (incipient ejection of spatter from pool) under keyhole mode laser welding conditions.*

## Accomplishments

We completed multiple activities including the following:

- *Modeling HED fusion.* We used a Monte Carlo model (Casino, public domain software) to assess energy deposition in various substrate materials and thicknesses as a function of e-beam accelerating voltage and beam size. Depending upon the material, its thickness, and number of layers, the AV can be used to control the location of the energy deposited, unlike a laser weld that deposits its energy on the surface, and requires vapor recoil-driven liquid surface depression

or solid state thermal diffusion to penetrate the material. The vapor recoil effect was effectively simulated by Sandia's GOMA code by introducing level set algorithms. Thermal analysis of the temperature distributions resulting from surface and volume heat sources demonstrated that, at the micron size scales of interest, temperatures are essentially steady state with very brief transients. Hence, the effect of nearby heat sinks is extremely important to understand how melting progresses.

- *Automating microEB welder.* After transferring our activities to a different SEM than the one we used last year, we were able to repeat and exceed previous size limits. However, despite having a digital interface to the SEM's operating system, the age of the device and its software idiosyncrasies left us unable to modify the stage and beam controls for flexible control of beam path and motion control.
- *Modeling cohesion/decohesion.* Our previous efforts indicated that at micron size scales, gaps can cause great difficulties in fusing parts together. Previously built surface-evolver models for spot welds in a sheet and across a gap between two bars were extended to a gap between two plates geometry. We investigated the consequences relative to retaining cohesion across the gap in a process model.
- *Modeling FIBDJ layer growth.* Experiments at determining optimal deposition beam parameters showed that wave-like effects occurred in the surface morphologies. This led us to investigate an initial, and then an improved, model for replicating these effects. The most recent two-dimensional model, which is extendable to three dimensions, employs a lattice gas cellular automata approach and indeed indicates that discontinuities in the surface cause propagating disturbances from a smooth fill.
- *Examination of substrate temperature on deposit properties and residual stresses.* We were unable to install the hot stage on the FIB; thus, we did not complete this portion of the project. We used a finite element model of the MEMS mechanical test vehicle to better understand the mechanical test results for samples produced at ambient conditions. These samples showed the large variability that is inherent in testing brittle materials. A large component of bending in the specimen's active gauge lengths partially explained the unexpectedly low properties. This was a result of using the alignment sidebars as the gauge lengths when the gap in the original tensile gauge was too wide to fill in a practicable manner. (The FIB's cutting capability was used to create grooves in the curved alignment sidebars around the original gauge length. These were subsequently filled with deposit to form a specimen that could be tensile tested.)

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# Magnetostrictive Elastomers for Actuators and Sensors

*52705*

*D. L. Huber, R. A. Anderson, J. E. Martin*

## Abstract

There is a need for soft actuators that have a much larger response than piezoelectrics and can respond in microseconds. Applications include artificial muscles in robots and stress/strain sensors based on permittivity or permeability changes. We theoretically demonstrated we can obtain large magnetostrictive effects from composites of magnetic nanoparticles in an elastomer, if the nanoparticles have been preorganized into chain-like agglomerates using magnetic fields. Such magnetostrictive elastomers (MEs) have a magnetic permeability that increases rapidly with compression so that they contract in a magnetic field, provided the particles are spaced by a soft matrix. This effect is especially large for nanoparticles where calculations show that the stresses should be comparable to that of human muscle. In both theory and experiment, we demonstrated the utility of our approach of magnetically structuring composites. We showed that the properties of the resultant ME can be controlled through an extraordinary range. We demonstrated the ability to systematically vary magnetostriction (both positive and negative), modulus, susceptibility, and electrical resistivity.

## Accomplishments

We made good progress in theory, modeling, and experiment designed to understand and evaluate the potential of MEs. These composites consist of magnetic particles in an elastomer. The magnetic particles are formed into assemblies by magnetic fields. The structures can be particle chains, particle sheets, or more complex three-dimensional particle assemblies, as described by Martin et al. [1]. In conventional magnetostrictive materials, the strain is due to the effect of spin alignment on atomic interactions within the magnetic material. The strain in MEs is due to the dipolar interactions between particles, and particle deformations make a negligible contribution to the effect. The degree to which these dipolar interactions can be altered by an applied field is strongly dependent on the structure of the particle assemblies, as described (in the isomorphic language of electrostriction) by Martin and Anderson [2].

We completed extensive experimental measurements of magnetostriction in a variety of composite materials having a range of particle loadings and structures. We discovered that, contrary to theoretical expectations, the magnetic field can be thought of as increasing the elastic modulus of these materials. We also demonstrated materials that exhibit negative magnetostriction. We showed that these materials can be designed to exhibit magnetoresistance of several orders of magnitude. We found that using complex magnetic field processing can greatly enhance magnetostriction in these materials, and that some of these materials exhibit significantly enhanced mechanical properties. We proposed and investigated theoretical models of field-driven structural instabilities in these composites. We developed a method of creating very high susceptibility nanocomposites and tested a number of these nanocomposites. As an additional activity, we prepared and measured hybrid composites made up of both microparticles and nanoparticles. The micron scale particles can be viewed as the magnetostrictive element, and the nanoparticles viewed as merely increasing the susceptibility of the matrix material.

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# Active Photonic Nanostructures

*58907*

*M. B. Sinclair, A. J. Fischer, P. G. Clem, J. G. Fleming, J. C. Barbour, N. S. Bell, G. S. Subramania*

## Abstract

Photonic crystals are periodically modulated dielectric structures that alter the flow of electromagnetic energy in many profound and potentially useful ways. Photonic band gaps, transmission resonances, restricted or enhanced densities of states, and modified spontaneous emission rates are all features of photonic lattices. In general, the photonic crystals demonstrated to date are entirely passive components: there are no means to alter, turn on, turn off, or modulate the unique photonic properties of these structures. The goal of this project is to integrate these passive photonic crystal structures with active materials to provide "active" functions such as optical amplification, lasing, light switching and steering, optical logic, chemical sensing, etc. In addition to greatly expanding the functionality of photonic crystals, incorporation of active materials will offer new opportunities for investigation of the novel physics arising from the strong modification of the photonic density of states and photonic band structures as they impact electron-photon interactions. This project is a collaboration between Sandia and Los Alamos National Laboratory and draws upon the demonstrated strengths of these institutions in photonic crystal structures, colloidal quantum dots, energy transfer in composite nanomaterials, nanomechanics, and self-assembly.

## Accomplishments

We made significant progress toward our goal of integrating photonic lattice structures with active materials. We investigated a number of different approaches toward the production of active photonic nanostructures, ranging from "top-down" fabrication to "bottom-up" self assembly.

(1) We fabricated InGaN light emitting diodes (LEDs) incorporating a two-dimensional (2-D) photonic crystal. The LED far-field emission pattern for photonic crystal LEDs is dramatically modified due to the presence of the photonic crystal, which scatters waveguided modes out of the semiconductor improving device efficiency. Photonic crystal LEDs fabricated at Sandia demonstrated a 1.5x increase in external quantum efficiency compared to unpatterned control LEDs.

(2) We developed three new technologies for nanoscale thin film integration toward integrated electronics and optoelectronics, including: a new method of contact printing that enables submicron (100–600 nm) deposition of metals and metal oxides; a method for integration of high refractive index (n = 2.5) $TiO_2$ films in electron beam patterned silicon waveguide structures for visible wavelength photonic crystals; and a method for integration of tunable refractive index (n = 2.2 to 2.4) $Pb(Zr,Ti)O_3$ films into nanoscale photonic crystal waveguide cavities to enable electrically tunable photonic crystal cavities.

(3) We developed and demonstrated a 2-D photonic crystal with a high Q (~1600) microcavity with a very low mode volume (~0.1–0.2 $\mu m^3$) and a wavelength that can be tuned by modifying cavity geometry. We developed a procedure for introducing nonlinear material, such as lead lanthanum zirconium titanate (PLZT), into the microcavity region using a method for making electrical contacts. We developed a prototyping technique based on multilevel electron beam lithography for fabricating 3-D photonic crystals in a layer by layer fashion. We fabricated a five layer woodpile lattice based on silicon with a lattice spacing as small as 550 nm with bandgap centered at 1.2 µm. We also fabricated a 3-D microcavity structure by introducing point defects in the fifth layer in a nine layer (~2 unit cell) woodpile. We developed a procedure to extend this into the visible frequency regime (lattice spacing 350–400 nm) using titanium oxide as a high index material. Introduction of active materials into these structure is useful in light emission and biosensing.

(4) We synthesized photonic colloidal structures that exhibit state-of-the-art optical properties. We attempted integration with fluorescent CdSe quantum dots. In addition, we fabricated novel core/shell nanoparticles consisting of a Au core and a $SiO_2$ shell. Such nanoparticles are expected to exhibit a higher refractive index than pure $SiO_2$ nanoparticles and thus be ideal building blocks for photonic crystals. We used self-assembly techniques to fabricate colloidal crystals from these core/shell materials. The resulting crystals exhibited bright interference colors, indicating the presence of highly reflecting photonic bands.

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# Electrochemically Switchable Materials for (Bio)Microfluidics

*67074*

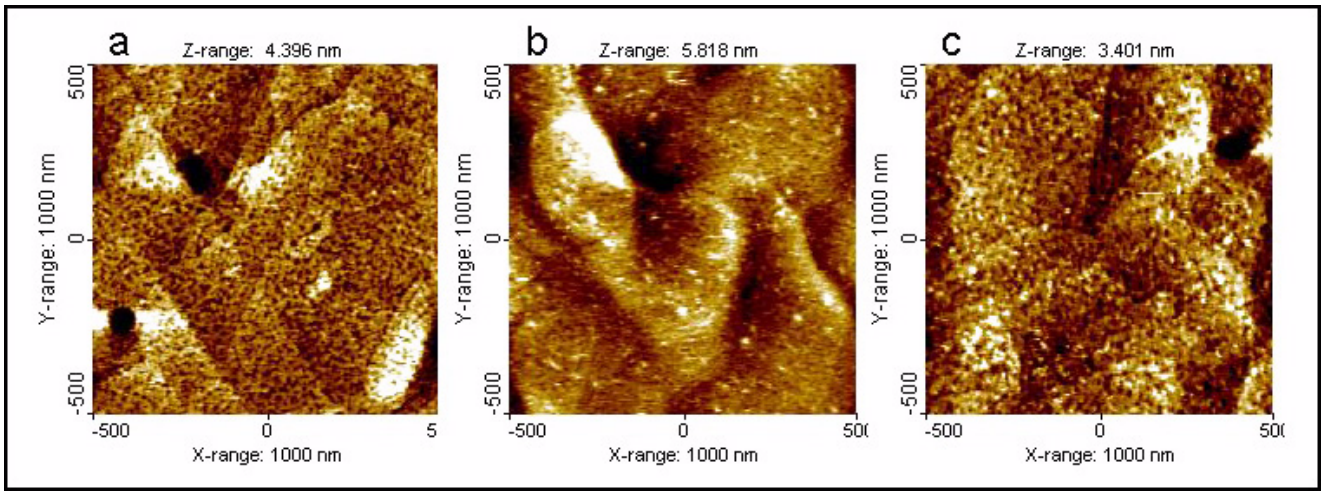
*K. R. Zavadil, B. C. Bunker, M. Farrow, D. L. Pile*


## Abstract

Homeland security needs require the development of a new generation of microfluidic devices that can concentrate, separate, and sense a variety of viral, bacterial, and toxic agents. The key enablers are functional materials capable of being programmed to perform a wide variety of operations on complex biological samples and that are compatible with nanoscale architectures. The purpose of this project is to address electrochemical means of controlling the transport of relevant species using binding or complex formation between host molecules and analyte guest molecules. We demonstrated the chemical attachment of a series of modified beta-cyclodextrin host molecules, synthesized and characterized in our laboratories, onto a variety of sensing platforms. These experimental platforms include electrochemical, nanogravimetric, and optical spectroscopic sensors for conducting quantitative measurements of the interaction between metallocene-based guest molecules and the host molecule. We synthesized a bioconjugate molecule, based on ferrocene modified biotin, and demonstrated that it will interact with the host as well as bind with the protein complex streptavidin. The streptavidin protein complex will further bind an immunoglobulin antibody to the surface attached host. Finally, we demonstrated electrochemical control and measurement of binding for this protein guest-host system. These accomplishments represent the completion of the first stage of development of a material system capable of exhibiting electrochemically switchable surface functionality for microfluidic sensing applications. These project goals are aligned with the development of multifunction biosensing devices by providing a class of materials and assembling technologies suitable for device integration and use.


## Accomplishments

We synthesized a variety of functionalized beta-cyclodextrin molecules for multidentate covalent bonding to Au surfaces. The primary variants were the length of the molecular chain used to tether the host molecule to the Au surface and the active chemical moiety that binds to the Au electrode. We investigated 1, 6, and 12 methylene unit chains that bind through thiol and sulfide links with Au electrodes. We demonstrated surface bonding using several electroanalytical techniques (voltammetry, square wave voltammetry, and electrochemical impedance) to probe binding activity, time-of-flight mass spectrometry to detect the host molecule




*Low force tapping mode atomic force microscopy (AFM) images of beta-cyclodextrin* (a) *on Au(111),* (b) *after binding ferrocene-biotin and streptavidin, and* (c) *after electrochemically ejecting the guest complex.*

directly, and atomic force microscopy to image the molecular distribution on atomically flat Au surfaces. Our experiments show that a beta-cyclodextrin with a 12 methylene tether based on a dialkylsulfide structure leads to compact host films that maintain electrochemically controlled binding activity for periods of greater than 12 months. The development of a stable material system results in durable electrodes that can be integrated into microfluidic systems.

We explored the interaction of a bioconjugate guest molecule with the surface-bound beta-cyclodextrin host. Biotin was functionalized with electrochemically active ferrocene. Using voltammetric techniques, we measured a formation constant of 1000 $M^{-1}$ for this complex. The surface concentration of the bioconjugate approaches unit activity or $4.4 \times 10^{13}$ molecules/$cm^2$, indicating saturated binding with the host. The bioconjugate shows a slow but measurable dissociation from the host in phosphate buffer solutions with as much as 20 percent of the population still bound to the surface after several hundred seconds in solution. Oxidation of the ferrocene ligands produces a nearly instantaneous release of the molecule from the surface. This demonstration of difference in release rate of the bioconjugate from the host is the basis of electrochemical control and switching of the surface function. This result represents a validation of the initial hypothesis central to this project and the accomplishment of one of our primary goals in this project.

We also demonstrated the ability of the bioconjugate molecule to serve as a tailored binding site for the surface attachment of relevant bioanalytes. We used nanogravimetry to monitor binding of the protein complex streptavidin to electrode surfaces through the bioconjugate molecule. We measured mass increases consistent with monolayers of streptavidin. Additionally, we used nanogravimetry to demonstrate the specific binding of an immunoglobulin antibody to ferrocene-biotin-streptavidin bound in the beta-cyclodextrin host on Au electrodes. Demonstrating specific binding activity creates the opportunity to conduct viable sensing operations in microfluidic applications.

The use of proteins raises concerns of nonspecific binding and resulting fouling or loss of function of the beta-cyclodextrin host. We synthesized several modified versions of the beta-cyclodextrin molecule that incorporate short polyethylene glycol ligands on the rim of the beta-cyclodextrin molecule. The addition of these substituents decreases the nonspecific binding of test proteins like bovine serum albumin and casein on the beta-cyclodextrin surface, as measured by nanogravimetry. These proteins are not retained at the beta-cyclodextrin surface and are easily removed with aqueous rinsing. This development of design strategies for resistance to biofouling coupled with durable electrodes and an electrochemically controlled materials system places this project significantly forward in our goal of concept integration into a microfluidic platform.

We are steadily progressing toward developing electrochemically switchable surface functionality that can be implemented into microfluidic-based sensors for various Department of Energy and Homeland Security missions. We are developing the scientific knowledge and capabilities necessary to design functional engineered surfaces and to define their viability at the micro- and nanoscale device levels.

## Other Communications

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# Modeling of Friction Induced Deformation and Microstructure

*67075*

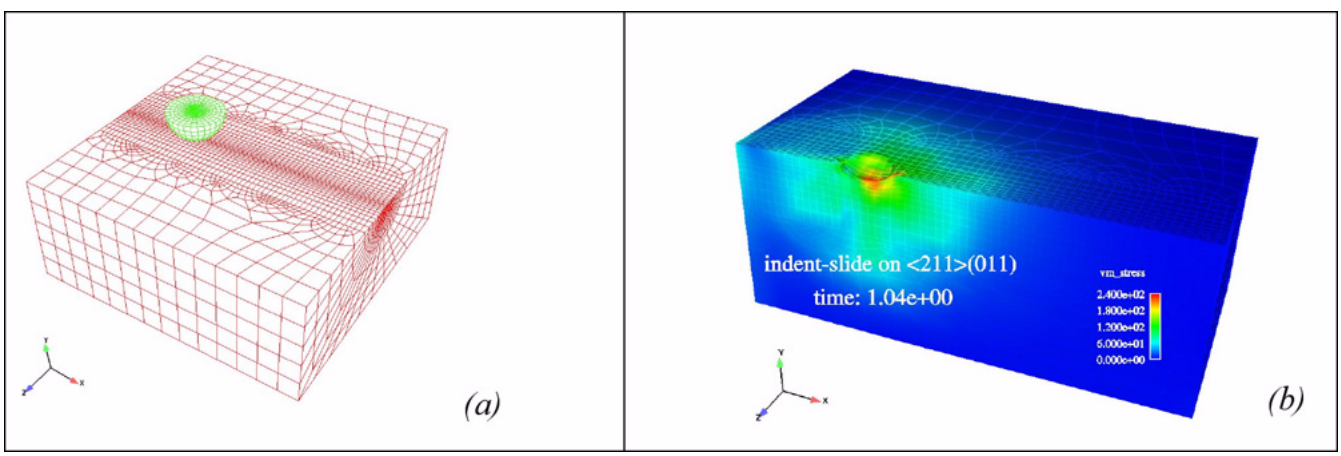
*S. V. Prasad, C. C. Battaile, J. R. Michael, R. E. Jones, E. B. Marin, J. J. Kelly, N. R. Moody, P. M. Gullett, D. J. Bammann*

## Abstract

Frictional contact results in surface and subsurface damage that can influence the performance, aging, and reliability of moving mechanical assemblies. Changes in surface roughness, hardness, grain size, and texture often occur during the initial run-in period and resulted in the evolution of subsurface layers with characteristic microstructural features that are different from those of the bulk. Recent research on single crystal nickel surfaces revealed the generation of low-angle boundaries and formation of nanocrystalline zones as a result of sliding contact. Bending of columnar grains has been observed in the case of textured materials such as electroformed LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) nickel. The objectives of this project are to develop models to predict the microstructural evolution and debris generation during sliding contact, and to validate the models with novel experimental techniques. Our models will focus on the plastic work in the near surface regions due to the Hertzian contact forces, and include work hardening recovery and microstructure evolution during sliding contact. We will extend the current focus on single asperity contact on single crystal surfaces to include polycrystalline materials. We will incorporate the influence of friction induced strains (elastic and plastic) on coating/substrate interface reliability. We will also validate our model by conducting friction tests on select microsystems materials and by analyzing the microstructures underneath the wear scars using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). These results will allow direct comparison with the model predictions and provide a more complete understanding of the friction and wear phenomena in regimes relevant to both micro- and macrosystems.

## Accomplishments

By employing techniques we developed in FY 2004, we evaluated the friction response of nickel single crystal surfaces along specific crystallographic directions. We studied the effect of normal applied load on recrystallized grain structures, and studied how these new structures in turn influence friction. Using an elastic Hertzian contact for a cylindrical-on-flat configuration, we calculated the resolved shear stress (RSS) for the various slip systems for the face-centered cubic lattice. The EBSD analysis showed that the material under the wear scar rotates about an axis that is in the




*Three-dimensional finite element simulation of the early stages of sliding contact using* (a) *a hexahedral mesh refined in the wear track to predict* (b) *deformation in the <211>(011). Colors represent von Mises stress.*

plane of the wear surface and normal to the wear direction, leading to the formation of dislocation cells. Once the rotation exceeds a critical value, the material recrystallizes. We performed finite element calculations to simulate material rotation during multipass sliding of a cylinder along various crystallographic directions on single crystal nickel surfaces, using a simplified crystal plastic material model. These calculations, when extrapolated to thousands of wear cycles, show near-surface material rotations in qualitative agreement with experiments. TEM analysis of wear scar sections revealed the formation of subsurface layers (with 5–10 nm grains) under certain loading conditions. Using the Raj and Ashby model, we estimated that substantial grain rotation during sliding contact can occur for nickel with a 5–10 nm grain structure. We believe that this transition from dislocation-mediated deformation in larger grained structures to a grain rotation deformation mechanism plays an important role in the frictional response of nanocrystalline materials. Using the Hysitron scratch test module, we made friction and nanoindentation measurements on electrodeposited nickel at micronewton loads. We performed three-dimensional finite element simulations of indentation onto five crystal surfaces using a material model for aluminum (as a prototype for nickel). The finite element analysis showed clear differences in the mechanical response on different crystallographic orientations.

## Refereed

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N.R. Moody, J.M. Jungk, M.S. Kennedy, S.V. Prasad, D.F. Bahr, and W.W. Girberich, “Small Volume Tribological Testing of LIGA Nickel,” presented and in *Proceedings of Fundamentals of Nanoindentation and Nanotribology III Materials Research Society Fall Meeting*, Boston, MA, 2004, CD-ROM.

## Other Communications

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J.R. Michael, S.V. Prasad, and B.L. Boyce, “Understanding Fatigue and Tribological Performance of Metallic Microdevices,” presented at the Materials Research Society Spring Meeting 2005, San Francisco, CA, 2005.

# Reversible Antibody Trapping for Selective Sensor Devices

*67076*

*D. L. Huber, S. M. Brozik, R. P. Manginell, G. D. Bachand, J. E. Houston*

## Abstract

Sandia has identified a need to develop compact sensors that can selectively detect low concentrations of a wide range of potential biological agents for homeland security applications. 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. This project investigates 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). We will use surface sensitive Fourier transform infrared (FTIR), interfacial force microscopy, and neutron scattering (at the Los Alamos Neutron Science Center) measurements to determine the following: (1) the extent and kinetics of antibody adsorption on activated PNIPAM vs. solution conditions (e.g., competition with other proteins), (2) surface concentrations and orientations of the antibodies, (3) antibody-antigen interactions vs. monolayer structure, and (4) 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. We will integrate the reversible protein trap into Sandia's highly mass sensitive shear horizontal surface acoustic wave 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.

## Accomplishments

The reversibility of the antigen/antibody complex was a major focus of the work in FY 2005. In FY 2004, we found that although we were able to adsorb and release tobacco mosaic virus (TMV), as well TMV specific Immunoglobulin G (TMV-IgG) individually, the complex of the two did not desorb from PNIPAM surfaces as expected. Both TMV and IgG are known to be stable well above the 50 °C at which we performed our adsorptions. TMV is famous for its incredible heat resistance and can be subjected to boiling water and remain infectious. Still there was some question as to whether the TMV/TMV-IgG may have some temperature sensitivity that is not seen in either individually. To test this hypothesis, we began studying release of the antibody/antigen complex as a function of temperature of antigen adsorption. If the TMV was adsorbed at below about 40 °C, we saw consistent release of the complex upon cooling of the substrate. We developed a new technique to allow the rapid collection of adsorption data. Determining adsorption isotherms has, until now, taken months of tedious data collection to produce statistically reliable data. We can now generate these data in a matter of hours. We redesigned our neutron reflectivity cell that will allow us to complete our neutron experiments. Finally, design of the microfluidics based sensor system is complete, and we are looking to begin fabrication in the coming months.

## Other Communications

D.L. Huber, B.C. Bunker, R.P. Manginell, T.S. Phely-Bobin, and G.D. Bachand, "Invited Lecture: Reversible Adsorption of Proteins on Poly(N-Isopropyl Acrylamide) and Implications to Biotechnology," presented at the International Conference on the Frontiers of Polymers and Materials, Cancun, Mexico, April 2005.

# Correlated and Comprehensive Analytical Techniques for Homeland Defense

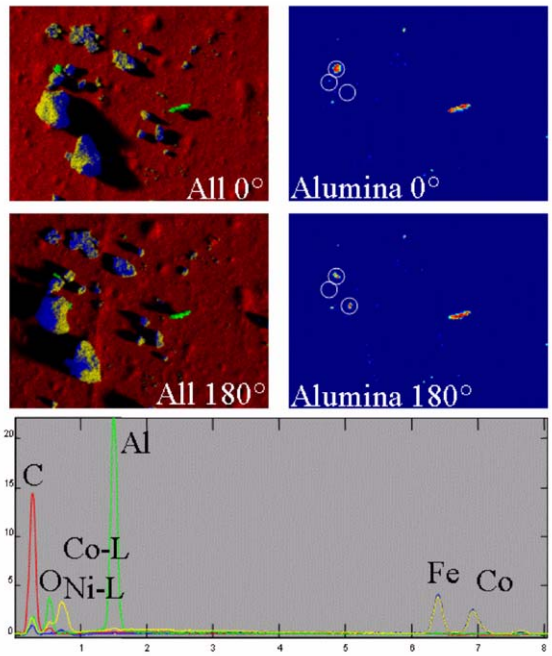
*67077*

*P. G. Kotula, J. A. Timlin, S. M. Sickafoose, A. J. Antolak, M. R. Keenan, B. L. Doyle, D. E. Peebles, D. R. Tallant, T. T. Borek III, J. A. Ohlhausen*

## Abstract

Identification of unknowns (biological/otherwise) to determine origin or potential threat requires multiple analytical tools covering molecular and elemental signals spanning both organic and inorganic materials. Additionally, each class of analysis may involve multiple independent techniques. While each individual analysis can be interpreted, the challenge arises in correlating all the disparate signals in a self-consistent and statistically rigorous framework, inputting data, and getting out knowledge. No tools currently exist that are up to this significant and important challenge. Sandia successfully developed a set of multivariate statistical analysis data mining tools called Automated eXpert Spectral Image Analysis (AXSIA) based on principal components analysis and constrained factor analysis. The specific approach to data analysis embodied by AXSIA has successfully been applied, independently, to x-ray microanalysis, x-ray fluorescence, and time-of-flight mass spectrometry. Significant challenges exist, however, to an even more general application of this approach to the simultaneous correlation of the analytical signals required for the comprehensive analysis of unknowns. This project will address (1) data acquisition/aggregation of multiple analytical signals from test and surrogate bioweapon materials covering a range of possible analyses, and (2) computational and algorithm development issues for the analysis of what are anticipated to be super data sets of more than 20 gigabytes.

We obtained a gamma-irradiated weaponized /non-weaponized simulant of anthrax (*Bacillus thuringiensis*) and analyzed the simulant material with multiple analytical techniques. In parallel, we characterized the range of analytical techniques with simple test specimens and with multivariate statistical analysis (MSA) tools that were developed to augment the AXSIA software. Additionally, and most importantly, we developed and tested new algorithms for single technique analyses. We eliminated one analytical technique based upon poor analytical results and unstable performance in general.




*Partial results of simulated annular x-ray detector. Images from 0 and 180 degree rotations. Red is the carbon substrate, green represents alumina particles, blue is the hard x-ray component from the Fe-Co (K lines only), yellow is the soft x-ray component from Fe-Co (both K and L lines), and black is the part of the specimen shadowed by the particles. The alumina particles circled show severe shadowing from the larger Fe-Co particles to different extents at the two rotations.*

## Accomplishments

We made significant progress in this research project: we analyzed data from new analytical techniques, we developed and implemented new MSA techniques, we obtained a new series of gamma irradiated test samples from Lawrence Livermore National Laboratory, and we hired a staff member.

We acquired and analyzed spectral image and series data from several analytical techniques. These included particle induced x-ray emission (PIXE), x-ray fluorescence (XRF), x-ray photoelectron spectroscopy (XPS), electron energy-loss spectrometry (EELS), gas

chromatography/mass spectrometry (GC/MS), visible spectroscopy (with hyperspectral scanner), and x-ray diffraction (XRD). We analyzed both simple test specimens and spore preparations. Additionally, we developed a new particulate analysis method in the scanning electron microscope (SEM) whereby an annular x-ray detector was simulated.

We developed new algorithms, tested them on several of the techniques above, and implemented them. The original MSA approach provided a spectrally simple view of large and complex spectra series and images. However, this is not always a good way to look at data where there are comparable signal levels in all of the channels. To solve this problem, we developed a new approach based upon maximizing spatial simplicity and applied this approach to the techniques above. Additionally, we applied equality constraints, based upon spectral derivatives of the intense components, to electron spectroscopy where the energy baseline shifts continually. In addition to the new algorithms, we made progress in adding new functionality to AXSIA, namely, the ability to counteract bias from the non-negativity constraint of multivariate curve resolution.

We obtained a new series of test specimens that contains gamma-irradiated *Bacillus thuringensis* (non-viable) spores grown under different known conditions, with and without weaponizing agents. We analyzed these specimens with both x-ray microanalysis and time-of-flight secondary ion mass spectrometry.

## Refereed

P.G. Kotula, M.R. Keenan, and J.R. Michael, "Tomographic Spectral Imaging with Multivaraite Statistical Analysis: Comprehensive 3D Microanalysis," to be published in *Microscopy and Microanalysis.*

## Other Communications

M.R. Keenan and P.G. Kotula, "Recent Developments in Automated Spectral Image Analysis," presented and in *Proceedings of Microscopy and Microanalysis*, Honolulu, HI, August 2005, vol. 11(Suppl. 2), pp. 36–37.

L.N. Brewer, P.G. Kotula, J.A. Ohlhausen, and J.R. Michael, "Spectrum Imaging Techniques Applied to Forensics Investigations," presented and in *Proceedings of Microscopy and Microanalysis*, Honolulu, HI, August 2005, vol. 11, (Suppl. 2), pp. 438CD–439CD.

P.G. Kotula and M.R. Keenan, "Multivariate Analysis of X-ray, Ion and Electron Spectral Images: From Surface to 3D Materials Characterization," presented and in *Proceedings of Microscopy and Microanalysis* Honolulu, HI, August 2005, vol. 11, (Suppl. 2), pp. 426CD–427CD.

P.G. Kotula, M.R. Keenan, and J.R. Michael, "Tomographic Spectral Imaging: Analysis of Localized Corrosion," presented and in *Proceedings of Microscopy and Microanalysis* Honolulu, HI, August 2005, vol. 11, (Suppl. 2), pp. 440CD–441CD.

# Development of High Energy Density Dielectric Materials for Integrated Microsystems

*67078*

*B. A. Tuttle, D. A. Hoke, M. A. Rodriguez, J. Cesarano III, E. L. Venturini, G. A. Samara, P. G. Clem, J. A. Voigt, V. K. De Marquis*

## Abstract

Next generation surety systems require compact, highly integrated microsystems to improve device functionality, miniaturization, and high-g reliability. Capacitors are by far the largest components in these systems; the greatest increase in volumetric device efficiency can be gained by reducing capacitor size. In the last three years, dramatic developments in dielectric science have been reported that can enable a 2x to 100x size reduction in capacitor size over state-of-the-art commercial materials. We intend to develop these new materials for high-field applications and integrate them into microsystems using direct write technologies. We are investigating two new dielectric materials families as alternatives to state-of-the-art dielectrics, which generally have a dielectric constant (K) of about 1100:

(1) Specially formulated lead lanthanum zirconate titanate (PLZT), K ~2500

While recently developed high Zr content PLZT capacitor materials exhibit twice the pulsed discharge energy of state-of-the-art $BaTi0_3$ materials, further improvements of 2x to 4x are anticipated via chemical preparation techniques and combinatorial composition optimization.

(2) Novel relaxor dielectrics, with temperature-stable K > 12,000

A small subset of relaxor dielectric materials, first reported in 2000, have temperature-stable relaxor dielectric characteristics that have not been observed previously in any other known materials. For these materials, $CaCu_3Ti_4O_{12}$, Ca:K(Ta,Nb)$O_3$, and (Pb,La) $TiO_3$, dielectric constants of 12,000 to 280,000 that are stable over a temperature range of greater than 300 $^oC$ have recently been measured but have not been integrated or evaluated at high electric fields. These materials could lead to a 10–100 fold, or greater, reduction in the size of pulse discharge capacitors.

Sandia is uniquely positioned to develop these microsystem enabling technologies due to the combined internationally recognized excellence of three different skill sets: (1) fundamental physics of ferroelectrics, (2) direct write technologies for chemically prepared ceramics, and (3) design and fabrication of surety microsystems.

## Accomplishments

We addressed the following four milestones:

(1) develop further processing procedures for polycrystalline novel relaxors (doped relaxor materials) and perform dielectric characterization,

(2) develop a chemical preparation (chem-prep) process for PLZT dielectrics,

(3) fabricate polycrystalline novel relaxors using direct write technology, and

(4) fabricate chem-prep PLZT dielectrics using automated aerosol spray deposition processes.

We chose $CaCuTiO_3$ (CCTO) as the primary novel relaxor dielectric for our investigation. Our work on CCTO under this project received international recognition. We investigated a wide range of p- and n-type dopants to enhance resistivity and breakdown strength. In collaboration with the NanoSIMS Center at the University of Washington, we combined $^{18}O$ and nanoSIMs (secondary ion mass spectrometry) studies and definitively determined the origin of the high dielectric constant behavior. We fabricated state-of-the-art polycrystalline novel relaxors with dielectric constants that range from 5000 to 160,000. We performed extensive testing on dielectric properties as a function of temperature, electric field, and hydrostatic pressure on Li and Ca doped potassium tantalum niobate single crystals, polycrystalline CCTO ceramics, and polycrystalline PLZT ceramics. We developed a chemical preparation process for PLZT dielectrics that has enhanced chemical homogeneity, improved dielectric properties, and lowered firing temperatures compared to conventional processes. A key accomplishment was densification at 950 $^oC$ that permits direct integration to low temperature cofired ceramic technology. Further, by combining polarization versus field modeling with phase diagram development, we determined what type of phases and phase transformation sequences with applied field lead to the best high energy density properties. Using aerosol spray deposition of newly developed antiferroelec-

tric/ferroelectric PLZT ceramic layers, we achieved a 100 percent increase in energy density compared to dielectrics presently used in Sandia systems.

## Refereed

R.K. Grubbs, E.L. Venturini, P.G. Clem, J.J. Richardson, B.A. Tuttle, and G.A. Samara, "Dielectric and Magnetic Properties of $CaCu_3Ti_4O_{12}$: Influence of Temperature, Pressure and Doping," to be published in *Physical Review B.*

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D.P. Williams, B.A. Tuttle, J.C. Cesarano, and M.A. Rodriguez, "Aerosol Spray Deposited PLZT Films," presented and in *Proceedings of Extended Abstracts 107th Annual Meeting of the American Ceramic Society Meeting*, Baltimore, MD, April 2005, vol. 1, p. 249.

# Nanolithography Directed Materials Growth and Self-Assembly

*67079*

*J. W. Hsu, J. A. Voigt, N. C. Simmons, C. M. Matzke, J. Liu, E. A. Holm, M. E. Coltrin, D. Scrymgeour, T. L. Sounart*

## Abstract

The goal of this project is to explore the use of nanotechnology to create templates on surfaces, which, in turn, determine the growth/assembly of inorganic nanomaterials for potential applications in bio/chemical sensing, optoelectronics, catalysis, and energy conversion. Our approach is "bottom-up" rather than "top-down," in which conventional optical and electron-beam lithography function. Instead, we adapt soft nanolithography techniques to create regions with different surface energy, resulting in selectivity in the locations that favor crystal nucleation. In addition, the crystal morphology can be changed through the addition of organic modifiers. Multistage growth can be employed to create complex nanostructures with exquisite control. 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. As a result of this project, we will gain new insight into crystal growth and self-assembly processes at the nanometer scale and produce nanostructures suitable for microsystems. The marriage between nanotechnology and bio-inspired crystal growth approaches shows promise for new, inexpensive, large scale manufacturing methods.

## Accomplishments

We furthered the optimization, development, and characterization of ZnO nanorod growth on Ag films. In combination with a multistage growth technique, we also achieved growth of complex ZnO nanostructures at predetermined positions on a surface. In addition, we developed solution stamping nanolithography, which combines soft lithography and sol gel processing for purely additive patterning of ceramics and metals. We also explored combining dip pen nanolithography (DPN) and microcontact printing, providing the advantage of parallel patterning and nanometer resolution to pattern surfaces.

Specific accomplishments and results include:

(1) Optimizing growth of ZnO nanorods on self-assembled monolayer (SAM) templated Ag surfaces. By using spinning coating to deposition molecules on the elastomer stamps, we can now routinely achieve excellent selectivity in crystal nucleation over a square centimeter sized area.

(2) Studying the effect of patterning on nucleation density. We observed that the nucleation density depends strongly on the pattern size as well as the distance from the nearest active regions. This indicates that diffusion of growth species plays an important role in our experiments. We initiated two theoretical studies to better quantify the experimental results.

(3) Combining multi-stage solution growth with micropatterned templating. This enables us to organize complex ZnO nanostructures on surfaces. We are interested in investigating the effect of morphology on materials and device properties.

(4) Understanding the effect of SAM endgroups on growth selectivity. We observed that ZnO nanorods preferentially grow on bare Ag surfaces and not on SAM covered regions, regardless of the endgroup chemistry (methyl or carboxylate). Scanning Auger results showed that while Zn does not bind to methyl endgroups of SAMs as expected for inhibition of ZnO crystal nucleation, Zn does preferentially bind to carboxylate endgroups of SAMs. Hence, preferential binding of growth species is not necessary for crystal growth.

(5) Performing various microscopy and spectroscopy experiments to study the optical properties of these ZnO nanorods. 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 a reducing atmosphere. In addition, we performed cathodoluminescence imaging. All rods are luminescent with approximately equal intensity.

(6) Patterning SAM on surfaces with combined microcontact printing (mCP) and DPN. DPN enables us to achieve smaller feature sizes than mCP. However, it is a serial process, and hence it is slow. We successfully deposited one type of SAM with mCP and

replaced it in selected regions with another kind of SAM. We showed that the different frictional properties of the two molecules results in observable image contrast. Our submission of a scanning electron microscopy image of complex ZnO nanostructures assembled via multistage growth on a Ag surface containing a micropatterned organic template won first prize in the "Science as Art" competition at the Spring 2005 Materials Research Society Meeting.

## Refereed

N.A. Chang, J. Richardson, P.G. Clem, and J.W.P. Hsu, "Additive Patterning of Conductors and Superconductors by Solution Stamping Nanolithography," to be published in *Small*.

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# Development of a Novel Technique to Assess the Vulnerability of Micromechanical System Components to Environmentally Assisted Cracking

*67080*
*D. G. Enos, S. H. Goods*

## Abstract

Microelectromechanical systems (MEMS) will play an important functional role in future Department of Energy 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, etc.) 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. The information gained will establish the appropriate combinations of environmental factors (defining packaging criteria), local stress levels, and metallurgical factors (composition, grain size and orientation) that must be achieved to prevent EAC.

## Accomplishments

The goal for FY 2005 was to refine the test technique and expand its capabilities. This work can be broadly grouped into two primary tasks: mechanical test system and crack detection sensor development, and sample refinement.

- Mechanical Test System and Crack Detection Sensor Development

We continued the development of a crack initiation/length sensor. We made additional attempts to construct a two-electrode probe consisting of gold pads, electrically isolated from the sample. We abandoned the use of an SU-8 photoresist in favor of paralene as the insulating layer. Unfortunately again, we could not avoid shorting of the sensors, and so the development of the crack detection sensor was put on hold until we identify a more promising insulator layer.

Due to significant technical hurdles to the development of an electrical or electrochemical based sensor to detect crack initiation, we focused on developing other techniques. We evaluated a number of potential techniques that might be capable of observing the evolution of environmental damage. We identified differential microscopy as being particularly promising. In this technique, a series of high resolution images of the surface are taken over time and quantitatively compared to one another through image subtraction or a similar post processing method. Subtle changes in the surface morphology that are not readily resolvable using traditional image analysis techniques can then be readily quantified. We are focusing on developing a technique based upon differential microscopy that can be applied to our atmospherically based environmentally assisted cracking studies. We designed and are constructing a test cell that allows optical analysis of the sample during exposure.

- Sample Refinement

We focused our initial experimentation on well-formed bars of material that were machined from a larger bulk material. While the composition of the material is similar to that of an actual component, the microstructure and physical properties may differ from a smaller, as-plated part for reasons described above. This year, we constructed samples from material in the as-plated state. Unfortunately, the flat bend bar samples we previously pursued were incapable of initiating EAC. Therefore, we produced new samples with induced defects—in this case, a single edge notch (root

radius of approximately 0.2 mm, machined using a fine diamond saw). The new sample geometry and associated modified mechanical test system provides a number of key features. First, much larger and better defined stress intensity can be reproducibly applied to the material in a well defined region (i.e., the notch root). Second, the new geometry provides a “defect” more representative of the types of things we would be concerned with for an actual MEMS part. Finally, it provides a large portion of material through which a crack might propagate, improving our chances of having a stable crack we can focus on for sensor development.

The experiments we performed using the new sample geometry successfully illustrated that EAC of the high sulfur-nickel can occur. This is something the previous sample geometry was unable to demonstrate. In addition, the rapid crack growth rate we observed clearly indicates that the cracking process is largely initiation controlled. This is likely the result of the sample geometry itself; once a crack has initiated, the applied stress intensity at the advancing crack tip is considerably higher than that which was applied at the notch root. As such, the crack is able to advance rapidly through the sample because the stress intensity does not fall off sufficiently with crack advance (and sample unloading) to arrest the crack. To test this hypothesis, we developed a mask that will produce as-plated samples with a notch/artificial defect with various dimensions, including one approaching the dimension of a crack.

# 3-D Optical Sectioning with a New Hyperspectral Deconvolution Fluorescence Imaging System

*67081*

*D. M. Haaland, G. S. Davidson, M. B. Sinclair, H. D. Jones, D. Y. Sasaki, J. A. Timlin, A. L. Slade, G. D. Bachand, G. D. Jones, T. L. Sounart, L. Nieman*

## Abstract

This project includes designing, building, and applying a novel hyperspectral fluorescence microscope for high resolution three-dimensional (3-D) optical sectioning of cells and subcellular organelles, 3-D monitoring of microfluidic processes, and investigation of molecular motors. We are developing data analysis methods to deconvolve the hyperspectral image data and to rapidly extract 3-D concentration distribution maps of all emitting species. The imaging system has many advantages over current confocal imaging systems: simultaneous monitoring of numerous highly overlapped fluorophores, immunity to autofluorescence or impurity fluorescence, enhanced sensitivity, and dramatically improved accuracy and dynamic range. The system can make detailed 3-D composition maps of targeted volumes at high spatial and temporal resolutions. Efficient data compression in both the spatial and spectral dimensions allows personal computers (PCs) to perform quantitative analysis of hyperspectral images of large size without loss of image quality. The new imaging system is enabling technology for numerous applications including the following:

(1) 3-D composition mapping analysis of multicomponent microfluidic processes such as mixing, dispersion, and chemical syntheses of materials,

(2) quantitative analysis and colocalization of molecular motors in three dimensions,

(3) examination of the binding of functionalized lipid vesicles to specific cells and the release and/or internalization of the vesicle contents, and

(4) understanding photosynthetic processes in wild type and mutant *Synechocystis cyanobacteria*.

This challenging project has already started to make Sandia a major player in advanced spectral imaging techniques for materials research and biotechnology.

## Accomplishments

We completed the construction of the 3-D hyperspectral imaging system and are using it to obtain excellent quality hyperspectral images. We continue to carefully characterize the diffraction-limited confocal imaging system. Software for operation and visualization of the data is now in place, and we significantly upgraded multivariate curve resolution software for extracting pure emission spectra and relative concentrations. We generated and tested C code for the spatial deconvolution of the hyperspectral images. We obtained 3-D hyperspectral images and analyzed the data for the following: (1) imaging of quantum dots in microfluidic channels, (2) imaging of molecular motors in three dimensions, and (3) examining the fluorescence detection ability and binding of functionalized lipids. In addition, we investigated supplementary applications with the new hyperspectral imaging system. For example, we imaged live *S. cyanobacteria*, live *Pseudomonas* bacteria in biofilms, and fixed rat basophilic leukemia cells to obtain preliminary data for a joint University of New Mexico/Sandia proposal to the National Institutes of Health. This exploratory research also formed the basis for another LDRD project on biofilms, and made the hyperspectral imaging system a key component of a new biotechnology-based Grand Challenge LDRD project at Sandia. We were able to obtain 3-D hyperspectral images and analyze the data for three applications: (1) imaging of quantum dots in microfluidic channels, (2) imaging of molecular motors in three dimensions, and (3) examining the fluorescence detection ability and binding of functionalized lipids.

## Refereed

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# The Science of Solutes: Transition Metals in LIGA Nickel

*67082*

*A. A. Talin, K. F. Janssens, D. L. Medlin, S. H. Goods, S. M. Foiles, E. Marquis*

## Abstract

The central aim of this project is to understand how transition metal (TM) solutes affect the microstructure and thermal stability of electrodeposits. While electroforming is adaptable to microfabrication, the metallurgy of electrodeposits is considerably more complicated than that associated with 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. We focused on Ni-Mn material, with a Mn concentration from 0 to 1 weight percent. The Mn acts to refine the grain size of Ni and results in a substantial increase in strength and hardness. In addition, Mn changes deposition texture and stabilizes the as-deposited grain structure by inhibiting recrystallization and grain growth.

Discovering how TMs accomplish these changes and optimizing 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 and Cu/TM samples, which vary in process history (deposition conditions, composition, etc.), using techniques ranging from the atomic (atom probe microscopy) through the mesoscale (transmission electron microscopy, scanning electron microscopy) to the continuum (x-ray diffraction). Computational models at all three scales will utilize experimental results and inform experimental efforts. Using this combined approach, we aim to optimize the material by understanding the science. This year we completed an extensive study of how Mn affects the microstructure, texture, and thermal evolution of Ni electrodeposits. In addition, we characterized the distribution of Mn and other impurities using a three-dimensional (3-D) atom probe, and proposed mechanisms to explain the thermal stability of Ni-Mn. Below 600 $^{o}$C, we observe grain boundary segregation in select boundaries, in agreement with our modeling. Above 600 $^{o}$C, we observe grain boundary pinning by precipitation.

## Accomplishments

We focused our experimental work on providing an accurate and detailed description of the annealing response of fine grained Ni and Ni-Mn electrodeposits (EDs) over a wide range of length scales to understand the role of Mn impurity in thermal stabilization of Ni. Using a combination of mechanical testing, x-ray diffraction, transmission electron microscopy, and 3-D atom probe, 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. Surprisingly, we observed that Mn concentration as low as 0.1 weight percent is sufficient to impart significant thermal stability to Ni electrodeposits that show little or no change in strength or microstructure after a one hour anneal at 500 $^{o}$C. Furthermore, the electrodeposits retain significant strength even at higher annealing temperatures, with no observed recrystallization up to 800 $^{o}$C. In addition, we found that the onset of grain growth in Ni-Mn above 500 $^{o}$C correlates with the increase in Mn mobility in Ni. We also observed grain boundary segregation at some boundaries, but no segregation at others. At higher temperatures, we observed precipitation of $Ni_3Mn$ in the vicinity of grain boundaries, suggesting a precipitate based thermal stabilization mechanism dominating above 600 $^{o}$C.

The remarkable thermal stability of Ni-Mn leads us to envision widespread application for this alloy in stampers for hot embossing and injection molding for compact disc/digital video disc (CD/DVD) manufacturing and nanoimprinting. Sulfamate Ni is commonly used to electroform stampers for CD and DVD production. One current challenge with these nominally pure Ni stampers is the loss of feature fidelity due to recrystallization. This deterioration means that new stampers have to be continuously fabricated. This problem is expected to worsen as feature dimension shrinks and feature density increases. To demonstrate the feasibility of using Ni-Mn for stamper production, we fabricated a nanoembossing Ni-Mn tool and successfully embossed several patterns. This work contributed to a patent application filed by Sandia.

It was speculated that twinning in Ni-Mn EDs might prevent recrystallization. To study this hypothesis, we developed a twinning model that takes a 3-D grain structure and forms twins in a physically and

crystallographically realistic manner. While our intent was to perform recrystallization simulations on these structures, subsequent experimental measurements indicated that the degree of twinning in Ni-Mn is very similar to that in Ni sulfamate, so twinning is not the thermal stabilization mechanism. However, a concurrent study suggested that the high degree of twinning in electrodeposited metals might enhance yield strength above and beyond the Hall-Petch effect. We therefore analyzed our twinned structures via polycrystal plasticity finite element modeling. We found that twinning does not significantly affect stress-strain response. Another suggestion was that segregation of Mn solutes can stabilize grain boundaries and prevent growth or recrystallization. Atomistic simulations showed that the magnitude of the solute-boundary interaction varies strongly with boundary type. This suggests that the degree of interaction of Mn with boundaries will depend strongly on the character of the boundary, as we observed experimentally. We also explored in detail the influence of transient solute concentration gradients on microstructural stability. We found that the local variation in solute concentration induces local dissolution of the precipitates. Consequently, the grain boundaries at those locations are not pinned, ultimately leading to anomalous growth. This is a potential mechanism for the observed coarsening in Ni-Mn above 800 °C. Finally, we developed a new technique for the determination of both boundary mobility and stiffness and applied it to an asymmetric sigma 7 tilt boundary in Ni. An unexpected feature of our results is the large variation of the stiffness with direction. This had not been previously demonstrated, and it highlights the complex, collective effects present in an evolving Ni-Mn structure.

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# Novel Gel-Based Technology for Sensors and Weapons

*67083*

*J. L. Lenhart, P. J. Cole*

## Abstract

A polymer gel is a physically or chemically crosslinked polymer that is highly swollen by solvent. Mechanically, the solvent creates a "soft solid," which is easily deformable yet still recovers from the deformation due to the elastic nature of the crosslinks in the polymer. Polymer gels offer potential for 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. A key to designing gel materials is to understand the role of polymer-solvent interactions on the gel structure and properties because solvent-polymer partitioning in a gel can be critical for the performance of gels in 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. This swelling-shrinking phenomenon can be exploited to manufacture sensors for biological and chemical contaminants. In other applications, it is undesirable to have a gel exhibit this phase separation, particularly when gel adhesion to a substrate is critical for the device performance. The objectives of this research are (1) to understand the role of the polymer-solvent interactions on the gel phase behavior, partitioning, microstructure, and properties; and (2) to utilize this knowledge to design gels suitable for implementation in various weapons devices and homeland defense applications.

## Accomplishments

In FY 2005, we focused on developing detailed knowledge relating to polymer-solvent interactions, phase behavior, microstructure, and properties in the gel. In particular, for the polybutiadiene gels, we explored in detail the impact of defects in the gel structure (entanglements, loops, and dangling chain ends). These defects had a profound impact on the mechanical, adhesive, and thermodynamic properties of the gel. This knowledge is essential for developing gels for alternative applications such as sensors. For sensor applications, we are exploring both polybutadiene and epoxy gel chemistries. The functional groups in these resins can potentially be modified in order to design receptor sites for chemical or biological agents. In addition, the wide array of monomer sizes and chemistries offers the potential to fine tune the gel properties for alternative applications. We started rheological and swelling experiments in order to understand the impact of subtle chemical changes on the gel microstructure and phase behavior. In order to develop techniques to exploit epoxy gels in sensors, we initiated experiments that focused on understanding the sensitivity and kinetics of the polymer-solvent phase transition in the gels.

## Refereed

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# Coupled Nanomechanical Oscillator Arrays for the Study of Internal Dissipation in Nanoscale Structures and Collective Behavior in Large Systems

*67084*

*J. P. Sullivan, N. A. Modine, D. W. Carr, J. R. Wendt, T. A. Friedmann, D. A. Czaplewski, W. Pan*

## Abstract

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. We developed a new approach to achieve this goal: the study of large arrays of coupled nanomechanical oscillators combined with theoretical calculations aimed at identifying atomistic mechanisms of dissipation. An important practical application of this research is the development of nanomechanical resonators for homeland security applications (e.g., sensors).

One objective of this project is to develop a means to measure oscillators at low temperatures where dissipation, due to tunneling states, and ballistic phonon transport become important. In addition, atomistic modeling will be performed on a system containing a known defect in order to directly model dissipation at small length scales. The fabrication of arrays 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 (2-D) ball-and-spring models, can be used to study disorder-induced localization in 2-D systems and stochastic resonance and non-linear effects in coupled systems. Specifically, we will examine localization of vibrational modes of the array at intentionally introduced defects. We will also examine the 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). Another key objective of this work is to identify the mechanisms that control internal dissipation in a variety of materials, ranging from amorphous systems to crystalline systems, and in mechanical resonator structures having dimensions down to the nanoscale. New experimental approaches will be developed to identify the nature of the defect relaxation processes that control dissipation.

## Accomplishments

Specific accomplishments include the first realization and measurement of 2-D coupled oscillator arrays consisting of tetrahedral amorphous carbon (ta-C) springs and tungsten island masses. We also designed and fabricated second generation 10,000 element arrays. These structures are suitable for physics studies of large systems of coupled oscillators. We completed numerical simulations on 20 x 20 arrays containing regions of disorder to represent a defect in an ideal lattice. The simulations revealed markedly different resonance characteristics for these arrays compared to ideal arrays, and these simulations will be tested by comparison to experimental systems.

We completed extensive internal dissipation studies on ta-C and discovered that internal dissipation in this material is controlled by dissipative defects, with an increasing concentration of defects having activation energies approaching or exceeding 1.5 eV. We further evaluated the characteristics of the defects that give rise to dissipation through experiments on both flexural and torsional oscillators (to evaluate defect relaxation under an applied uniaxial and pure shear stress state). This is the first study to characterize the internal dissipation and the nature of the dissipative defects in this material. The construction of the variable temperature microelectromechanical system (MEMS) oscillator test apparatus in this project represents a new capability for Sandia. In order to contrast internal dissipation in a purely amorphous carbon material compared to a crystalline material, we performed dissipation studies on polycrystalline diamond. These measurements revealed that very different defect mechanisms are operative in this system, and that the nature of the internal dissipation depends on details of the microstructure and doping of the diamond films. Using high resolution transmission electron microscopy, we successfully and clearly revealed microstructural differences between different films; and we showed, for the first time, the impact of microstructure on internal dissipation in polycrystalline diamond. As a side benefit, we were able to demonstrate the highest

quality factors ever observed for cantilever-based MEMS resonators: > 100,000 at room temperature. Lastly, given our focus on dissipative defects, we redirected our theoretical simulations towards understanding the properties of a specific defect: the split interstitial in crystalline silicon. This should enable direct comparison with experiments on single crystal Si oscillators.

The significant technical accomplishments we achieved this year produced many auxiliary benefits. For example, as a result of this work, Sandia established a new capability for characterizing internal dissipation in MEMS materials. A direct outcome of this effort is a new Basic Energy Science project funded by the Department of Energy focused on coupled oscillator arrays and their use in fundamental studies of internal dissipation.

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# Precisely Controlled Picoliter Vessels with Rapid Sample Preparation for Trace Biotoxin Detection

*67085*

*R. V. Davalos, B. A. Simmons, E. S. Lee, D. Robinson*

## Abstract

In existing microfluidic devices, diffusion and nonuniform flow velocities often cause unwanted mixing and dilution resulting in broad, nonuniform concentration profiles and reaction rates. When only trace amounts of sample material are available, as is often the case in forensic analysis or security screening, these effects can seriously degrade device performance. Electroosmotic flow is the state-of-the-art way to achieve uniform velocity profiles, but it requires bulky and dangerous kilovolt power supplies that largely defeat the purpose of microfluidic devices. To achieve improved control of mass transport and sample volume in microfluidic devices, we are studying the application of 10 micron scale artificial lipid vesicles that can load analytes or reactants, contain them in fixed volumes during reaction or transport, and release them or combine them with another container at a later time. We are developing new preparations of vesicles that can be integrated into microfluidic devices in a "just add water" mode: when analytes are added to a microfluidic channel, they encounter a dry lipid film built into the device that seals around them upon contact. The vesicles must then be sturdy enough to withstand pressure-driven or electrokinetic manipulation but still be able to become porous or to fuse with other vesicles upon electrical stimulation. We are also developing microfluidic test structures and instrumentation that allow us to devise optimal structures and protocols for handling the vesicles and to demonstrate their utility. Our goal is to produce a complete device prototype in which an analyte-containing liquid is introduced into a device, trapped, reacted, transported, and released autonomously. Such a demonstration will provide the building blocks for compact devices that can sensitively analyze picoliter volumes (or larger samples, broken into picoliter volumes without dilution) of samples in homeland security or nonproliferation investigations.

## Accomplishments

This research produced the first demonstration of controlled vesicle electroporation in an on-chip microfluidic platform. 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. We made clear progress in each of the three major areas of investigation: vesicle synthesis, vesicle loading and unloading by electroporation, and design of plastic microfluidic vesicle-handling devices.

We developed a new method for preparing vesicles that is compatible with field applications in which dried vesicles on a stored microfluidic chip are rehydrated immediately before use. We can quickly and reliably make picoliter vesicles while producing far less hazardous waste than previously published approaches, control vesicle sizes and shapes by adjusting chemical conditions on the inside and outside, and trap fluorescent markers inside vesicles. Using a prototype microfluidic device, we were able to immobilize individual vesicles on low-pressure ports, electrochemically open and close them, and monitor their porosity by measuring electrical current passing through the vesicle walls. Improvements we made to the test platform include electronics and software to mitigate electrochemical noise and increase sensitivity and integration of a pressure transducer. We also developed a new two-level plastic chip for vesicle manipulation that includes a filter and two stations for trapping and opening vesicles, the first at Sandia with 2 micron features and embedded electrodes.

These successes all point us toward achieving our goal: a microfluidic sample management platform in which vesicles are used as picoliter reaction containers that are autonomously moved and manipulated with electrical feedback through a number of sample loading stations.

## Refereed

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# Developing the Foundation for Polyoxoniobate Chemistry: Highly Tunable and Exploitable Materials

*70799*

*M. D. Nyman, T. M. Alam, M. A. Rodriguez*

## Abstract

In this project, we are continuing to expand the new field of polyoxoniobate chemistry that we initially discovered in 2002. This work has potential applicability in virus mitigation and pathogen binding both in vitro and in vivo. We structurally characterized a variety of new compounds that are representative of this emerging class of materials. These studies revealed what is possible and what is more challenging. We are now facing the challenge of expanding the field beyond its current boundaries by first understanding the reason for these limitations. Some of these hurdles include poor solubility of compounds and limited ability to use different counterions. We are investigating the role of the counterions through nuclear magnetic resonance (NMR) studies. We developed a method for disrupting insoluble frameworks while retaining the polyoxoniobate clusters intact.

## Accomplishments

The most important accomplishment this year is that we developed a benign and generally applicable method to disrupt and dissolve insoluble frameworks of polyoxoniobate anions. This is of major importance for continued expansion of this new field of study. Many polyoxoniobate phases that we discovered are insoluble or poorly soluble alkali salts, which in many cases limit carrying out further chemistry with these phases. Furthermore, we are limited in investigating applications that involve dissolution of the polyoxoniobate cluster. We discovered that addition of a surfactant detaches the anionic clusters via disruption of the insoluble framework. Furthermore, the surfactant-cluster phase that results from this process is soluble in less polar solvents such as alcohol. This now allows us to carry out further chemistry with these anions in non-aqueous solutions, which is necessary to expand the range of attainable polyoxoniobate compositions and geometries. Applications that might benefit from mobilizing the insoluble polyoxoniobate network include those related to their antiviral or pathogen binding capabilities. Our antiviral studies will be expanded to include these phases that had been previously deemed insoluble in biological-type media.

We also expanded our series of alkali salts of polyoxoniobates to include lithium salts. One of the lithium salts is a three-dimensional (3-D) arrangement of polyoxoniobates, which rounds out our range of dimensionality of phases from 0- (soluble ions) though 3-D. These lithium salts were challenging to obtain due to the poor solubility. To obtain these in a more controlled fashion, we developed a synthetic trick of starting with the more soluble rubidium salt precursors in a solution containing excess lithium hydroxide. Since we can form alkali heteropolyniobate salts of lithium, sodium and potassium, but not rubidium and cesium, we initiated a series of NMR studies to understand the reason for this, and to understand how we might get around this limitation. This is extremely important because it has been shown in other polyoxometalate materials that the counterion plays an important role in templating cluster formation. Therefore, using alternative counterions provides an opportunity for the development of new phases. Also in these NMR studies, we continue to develop our understanding of the role of protons in the formation of heteropolyniobates. We developed a synthetic method to introduce these protons into a polyoxoniobate salt in a controlled manner.

We synthesized our first polyoxoniobate phase featuring an organic countercation, which opens possibilities for new geometries and compositions, and possibilities for carrying out further chemistry in non-aqueous media. Additionally, this may lead to the development of new porous materials since organic cations may be removed by chemical or thermal methods and leave empty pores behind.

We also made inroads into polyoxotantalate chemistry this year by discovering methods to dissolve monomeric tantalate precursors and convert them to a polyanion in a controlled fashion. This is important because the big hurdle to polyoxotantalate chemistry is the poor solubility and high reactivity of tantalates in caustic reaction media.

## Refereed

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# Diatoms as Molecular Architects

*79821*

*B. A. Simmons, E. D. Spoerke, P. Lane, T. W. Lane, J. A. Zimmerman*

## Abstract

We are using a three-pronged approach to develop bioinspired hierarchical multiscale silicates that are based on the silica polymerization process that occurs in diatoms. Current bioinspired methodologies for the realization of self-assembled nanomaterials are adequate for certain applications, but a major breakthrough is needed if these materials are to achieve everyday use. One of the most promising fields of nanotechnology lies in the control of biologically self-assembled materials, especially those involved with silica and other ceramics such as hydroxyapatite. Diatoms are eukaryotic algae that are ubiquitous in marine and freshwater environments. They are known to be the dominant form of phytoplankton and are critical to global carbon fixation. Diatoms produce intricate silica structures, known as frustules, during cell division and reproduction that are of two basic types: pennate and centric. They do this in a multistaged and genetically controlled division pathway that includes silica transport, silica polymerization, and structure formation. We propose to understand and harness the organized silica deposition process that exists in diatoms in order to achieve biologically controlled three-dimensional self-assembly of complex nano- and mesoscale structures.

This project combines our expertise in materials chemistry, biotechnology, and computational modeling in order to elucidate the fundamental mechanisms by which these organisms produce these fault-tolerant and robust structures. We are accumulating and utilizing the chemical and proteomic results produced in these focus areas to produce both natural and synthetic materials that allow us to gain insight into the critical factors that determine when, how, and where the diatom replicates the silica frustule. We will couple these efforts with a computational evaluation of biologically controlled silica deposition and emergent intercellular phenomena to identify the rudimentary design rules by which these organisms operate. The successful completion of this research will enable advances in the fields of coatings, responsive materials, and catalysis.

## Accomplishments

In the materials science effort, we demonstrated that dendrimers and proteins induce silica polymerization at room temperature in buffered solutions that mimic the silica deposition environment found in diatoms. We also photolithographically patterned dendrimers on surfaces and showed that the resultant silica structures are defined by that pattern. We obtained three synthetic peptides that will be used for further exploration of the exact conditions that are optimal for the silica deposition process.

We also demonstrated a biomimetic approach to the self-organization of silica using microtubules. We demonstrated the first step of this process with the creation of artificial microtubule organizing centers. This approach gives us unprecedented control over the orientation of microtubules on functionalized silica microspheres. We also developed methods to polymerize these microtubules in functionally varied segments such that either the “plus” or “minus” end of the tubes are rich in biotin. These tubes may then be readily organized around silica microspheres functionalized with biotin’s reactive counterpart, streptavidin, to form organized assemblies.

In the biotechnology effort, we obtained and cultured six different species of native diatoms to provide us with a wide range of genetic diversity. In order to understand the genomic/proteomic pathways responsible for silica polymerization, we characterized the cycle of silica uptake, frustule formation, and cell division. This information will provide the framework that will allow us to track this morphogenic process through microscopy, proteomic analysis, and genomic analysis. We observed a correlation between the silica uptake into the cell and the resultant cell growth/division at a specified time. Through dye uptake experiments and confocal microscopy, we confirmed the time period during which frustule encoding genes were turned on and the proteins responsible for silica polymerization were expressed.

# Novel Mechanisms of Nanomechanical and Transmembrane Actuation

*79823*

*K. Leung, C. J. Medforth, J. A. Shelnutt, K. R. Zavadil, M. E. Chandross, S. L. Rempe*

## Abstract

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 including synthesizing robust, integrated nanosized devices, manipulating nanoparticles on surfaces, and 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 creating "nanotweezers" by inducing motion in the tightly bound porphyrin ligands and causing nanomechanical motions that can be amplified via specifically tailored structures attached to the porphyrin. Tightly bound metal-ligand interactions are also suspected to participate in the actuation (i.e., opening/closing motion, or "gating") of biological transmembrane ion channels. These channels transmit or block electrical signals in response to gating events that are strongly correlated with channel conformation and selective ion binding. X-ray data exist for the closed KcsA configuration, but these 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 employing an integrated research approach. We will use computer modeling assisted synthesis, x-ray crystallography, nuclear magnetic resonance (NMR), Raman, ultraviolet-visible absorption, and quantum spectral simulations to obtain a clear understanding of nanomechanical motion in robust photo- or chemo-triggered porphyrin. We will develop theoretical techniques to treat actuation and nanomechanical motion, using porphyrins as test cases, and extend them to correlate metal ion (e.g., Na, K, Tl, Rb) size-induced conformational changes in ion channels with large scale channel-opening mechanical motion. 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 delivery of biosensors.

## Accomplishments

- We began modeling the potassium channel (KcsA) selectivity filter.

Starting from the published low temperature crystal structure of the KcsA potassium ion channel protein, we used classical force fields to search for minimal structures that contain the critical "selectivity filter" responsible for selective potassium passage. We predict that potassium ions and water molecules occupy alternating sites in the filter, in agreement with crystal structure data. However, these force fields predict incorrectly that Rb+ and Cs+ ions are ejected from the filter. To test these force fields, we have and will continue to apply quantum mechanical calculations to treat many-body interactions and polarizability effects. We built a dedicated Intel computing environment to continue our studies using the Gaussian code.

- We completed joint experimental and theoretical analyses of model compounds related to the conformational changes in porphyrin based molecular switches.

Our initial study of model compounds related to these switches or nanotweezers shows that, in porphyrin model systems, the expansion of the low-spin nickel(II) ion to the high-spin nickel ion by axial ligation to amines has a dramatic affect on the stability of the different conformations, even though the Ni-N distance increases by only about 0.1 Angstrom. Low-spin nickel ion complexes strongly favor the ruffled conformation, whereas the high-spin nickel ion favors more planar/domed conformations. These findings confirm the underlying mechanism we proposed for the opening and closing of the nanotweezers. For porphyrins, which are engineered to be very ruffled by adding bulky peripheral substituents, a point is reached where the porphyrin conformation cannot be switched because the energy required to flatten and dome the macrocycle is much larger than the ligation energy.

- We built a multiprocessor 64-bit workstation with the Amsterdam Density Functional code installed. This will enable modeling the structures and electronic

properties of porphyrins at a higher level than is currently available. We completed a theoretical study of transition metal porphines adsorption on flat Au(111) surfaces. We found that Mn(II) porphines (MnPs) exhibit a 0.8 eV, site-specific binding energy when adsorbed flat on Au(111), and undergo significant charge transfer and spin-state changes. These changes can be partially reversed by applying a 0.7 eV/Angstrom electric field, which switches the oxidation state in adsorbed MnP from Mn(III) back to Mn(II) and creates a substantial (0.13 Angstrom) increase in the Mn-N distance. This increase is similar to that which causes the large scale tweezer-like motion for the bridled nickel(II) porphyrins discussed earlier. We propose that depositing manganese porphyrins or expanded porphyrins on electrodes may indeed be a viable route to "smart" composite materials and nanomechanical devices.

Our entire cross-disciplinary team of theorists contributed to this work. We apply the density functional theory $+U$ (DFT$+U$) method (successfully used to study colossal magnetic resistance materials but applied to transition metal porphyrin molecules for the first time), parameterized using our Gaussian quantum chemistry calculations. Classical force field calculations give estimates of the dispersion forces between porphine and gold surfaces.

- We began work on scanning tunneling microscopy.

We began work on molecular scale imaging of bridled porphyrins and simple unsubstituted porphine molecules on metal surfaces. The primary technique has been scanning tunneling microscopy both in ambient conditions and in solution. Imaging takes the form of low tunneling current monitoring at moderate bias potentials. Previous work showed that porphyrin lattice resolution is possible for adsorbed layers in solution. We attempted to obtain images of two planar porphyrin molecules, a Pt porphyrin and a Ni porphyrin substituted with ethyl and methyl groups at its perimeter. Adsorption was conducted from various organic solvents onto Au(111) and highly ordered pyrolytic graphite surfaces. Imaging was not yet successful; we modified the microscope to improve its sensitivity in lower current imaging. We implemented porphyrin force fields into the Sandia LAMMPS molecular dynamics code. We are applying porphyrin force fields developed by members of our team to study the stability and energetics of open and closed nanotweezer structures and the transitions between them.

## Refereed

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# Carbon Nanotube Sorting via DNA-Directed Self-Assembly

*79824*

*A. L. Frischknecht, M. G. Martin, D. Robinson, K. Leung, S. L. Rempe, A. A. Talin, P. M. Dentinger*

## Abstract

Single-wall carbon nanotubes (CNTs) have shown great promise in novel applications in molecular electronics, biohazard detection, and composite materials. CNTs 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 CNTs according to their geometric/electronic properties. Very recent experimental work published in *Science* demonstrated that single-stranded deoxyribonucleic acid (DNA) will bind to CNTs, 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. We are investigating DNA/CNT binding and its dependence on DNA sequence and the solution environment. DNA/nanotube hybrids have been characterized by a variety of techniques, including DNA melting experiments, adsorption spectroscopy and atomic force microscopy. Ultraviolet-visible (UV-vis) spectroscopy has been used to determine the relative affinity of a variety of nucleotide monophosphates for CNTs in aqueous solution. We are using molecular modeling and simulations to elucidate both DNA/CNT and ribonucleotide monophosphate/CNT binding. Using ab initio and hybrid quantum mechanical/molecular mechanical (QM/MM) calculations, we are exploring the effect of nucleic acid bases on nanotube electronic properties, a crucial step for developing electronic and sensor applications based on CNTs. Ultimately, this work will impact Sandia's mission in science based materials processing, novel sensors for homeland security, and control of nanosystems.

## Accomplishments

We made significant progress in FY 2005. We focused on building understanding of the many interactions in aqueous DNA/CNT solutions. We began by investigating the force fields available in the literature to simulate DNA/CNT interactions, and implemented these in the Monte Carlo code Towhee. We also modified the code to create starting coordinates for any single-walled carbon nanotube given the chiral vectors and the number of unit cells desired, and to generate starting configurations for the ribonucleotides of interest as well as DNA of any specified length.

The major effort for the year was a coordinated computational and experimental investigation of the interactions between individual nucleic acid bases and nucleotides, with CNTs as a starting point to understanding the interactions of DNA with CNTs. From UV adsorption measurements, we determined the relative affinity of seven different nucleotide monophosphates for CNTs. From the experiments, the following affinity series can be deduced as a qualitative measure of the interaction between nucleotides and nanotubes: adenosine > guanosine > xanthosine ~ inosine > thymine > cytosine > uridine (all monophosphates). This is interesting because the series falls in approximate decreasing order of the size of the bases. We are performing molecular dynamics simulations of the same set of nucleotides we studied experimentally. These simulations include water and counterions and will determine the enthalpies and, hopefully, also free energies for nucleotide adsorption onto CNTs to compare with the experimental results. Preliminary results indicate that an adenosine monophosphate does indeed bind preferentially to a CNT in aqueous solution.

Ab initio calculations of geometries and energies of small molecules, including nucleic acid bases, near tubes are in progress. We calculated the interaction energy of water with the exterior of the CNT and found a similar value to that for water inside a CNT. The calculation of dispersion effects on this energy is in progress, as well as calculations of the interaction energies between a model (6,0) tube and the nucleic acid bases thymine and uracil. The ab initio binding energies will benchmark classical force field results, and both should yield information on how changes in the hydrophilic/hydrophobic character of the bases alter interactions with CNTs.

Finally, we performed plane wave-based (quantum mechanical) density functional theory (DFT) calculations of various small molecules adsorbed to CNTs and

graphene sheets. Plane wave-based DFT calculations are uniquely suited to distinguishing metallic tubes, such as the (6,0), from insulators such as the (8,0) tube. With this method, we can assess whether the nonpolarizable classical force fields we use are qualitatively correct in neglecting electronic structure differences. We find that the conductivity has only small effects on the adsorption energies. The DNA bases (uracil and thymine) and even the highly dipolar NaCl clusters exhibit similar adsorption energies (~1.5–4 kcal/mol) on both types of tubes. This justifies the omission of tube electronic characteristics in the classical simulations.

# Next Generation Contact Materials for High Reliability Microsystems Devices

*79825*

*P. T. Vianco, M. T. Dugger, T. M. Mayer, J. A. Knapp*

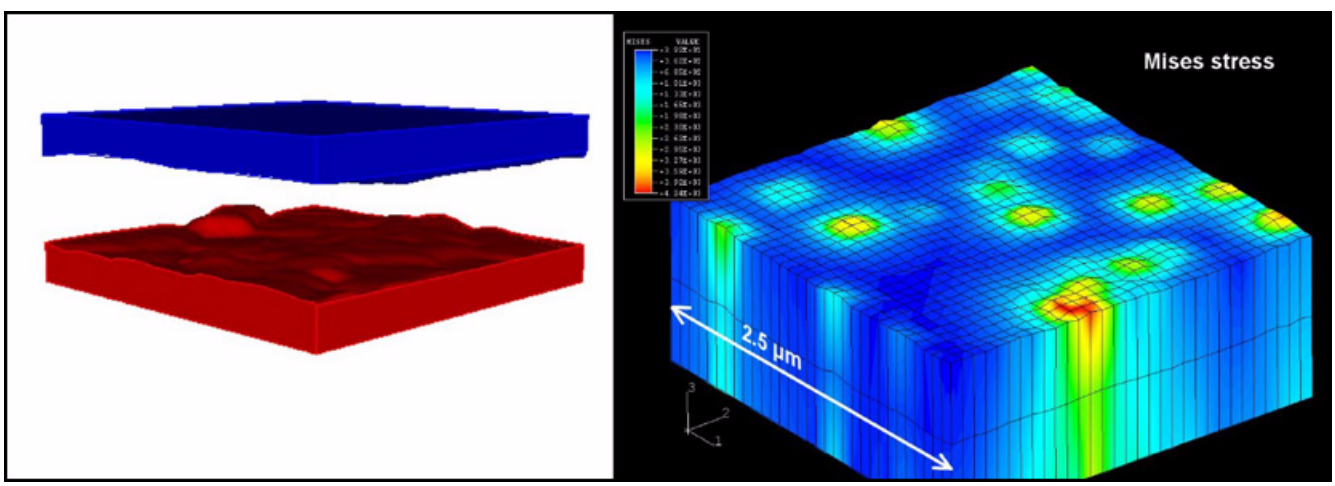

## Abstract

Dynamic electrical contacts are essential to weapon 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. The purpose of this project is to examine the effects of contact force, electrical current, and operating environment on contact resistance and adhesion properties in order to correlate the resistance and adhesion behaviors of fundamental degradation mechanisms such as microwelding, surface oxidation (kinetics), contamination build-up, and changes of contact material properties.

We conducted electrical contact resistance and adhesion tests of intermixed metal thin films of Au-Co and Au-Ge using a modified nanoindentation apparatus. We performed annealing in high vacuum in order to circumvent large scale surface oxidation observed with air annealing. Electrical contact resistance tests showed that the annealed films had only slightly higher contact resistance than pure Au-Au contacts and also had slightly lower adhesive pull-off forces in some cases. Auger electron spectroscopy depth profiling confirmed that only small amounts of Co and Ge diffused to the surface in vacuum annealing. We performed preliminary experiments on Au/titanium nitride film contacts that showed low pull-off forces and an unexpected increase in film contact resistance with decreasing thickness. We identified a suitable test structure to investigate new electrical contact films and evaluated it with Au-Au and Au-Pt films. We obtained surface topological data from Au-Ge multilayer films to establish the asperity geometry for the contact resistance computational model. We also measured thin film mechanical properties as input data for the model.


## Accomplishments

We conducted electrical contact resistance and adhesion tests of intermixed metal thin films using a modified nanoindentation apparatus. This apparatus simultaneously measured applied force, displacement, electrical contact resistance, and adhesive pull-off force in an inert, flowing nitrogen environment. We applied mechanical and electrical loads that we selected as representative of microscale electrical contacts. Bulk wafers of the intermixed Au-1wt.%Co and Au-1wt.%Ge films were cleaved into 1 cm x 2 cm samples.




*Surface asperity computational model.*

We brought these flat samples into contact with a 1.6 mm diameter Au-coated $Si_3N_4$ sphere under controlled loading conditions inside the experimental apparatus.

We observed that elevated temperature film annealing of the AuGe and AuCo film stacks in air caused significant surface oxidation to occur. Although the surface oxidation potential provided a driving force for intermixing of the elemental constituents, the oxidation degraded surface conductivity to such a degree that it was impossible to obtain a stable electrical contact even at highly increased normal loads.

We performed annealing in high vacuum in order to circumvent large scale surface oxidation. The vacuum-annealed AuCo and AuGe film surfaces did not show visible signs of surface oxidation. Electrical contact resistance tests showed that the annealed films had only slightly higher contact resistance than pure Au-Au contacts and also had slightly lower adhesive pull-off forces in some cases. Auger electron spectroscopy depth-profiling confirmed that the driving force for the diffusion of Co or Ge to the film surface in vacuum was not high enough to sufficiently intermix the film stack.

We identified several potential dichalcogenide layers as alternatives to the multilayer thin film technique. We performed preliminary experiments on Au/titanium nitride film contacts and observed low pull-off forces in testing. However, an unexpected result was an increase in film contact resistance with decreasing thickness.

We identified a suitable test structure with which to evaluate new electrical contact films. The structure provides the versatility to change one or both contact materials. Contact resistance can be measured directly, both hot and cold switch, for tens of millions of cycles. We evaluated the test structure with Au-Au and Au-Pt films. We developed a second test structure to locate micron sized regions so that, in conjunction with the apparatus introduced above, it will be possible to examine the surface morphology of the actual point of contact.

We began the computational modeling effort and set up the finite element mesh for surface asperities modeled after those measured on the candidate thin films. We obtained the mechanical properties of the multilayer thin films by nanoindentation and fed these into the numerical model. We performed test runs to establish a baseline mechanical response code for the model. Although we realized success for the Si baseline material, the softness of the Au-based thin films presented us with computational difficulties.

## Other Communications

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# Controlled Fabrication of Nanowire Sensors

*79826*

*F. Léonard, B. A. Simmons, L. L. Hunter, N. S. Bell, P. M. Dentinger, T. J. Boyle, A. A. Talin*

## Abstract

The purpose of this project is to apply an integrated multidisciplinary approach, including nanowire synthesis, device construction, and theoretical modeling, in order to develop a new class of sensors with nanowires as the active elements. This new class of sensors is based on dramatic changes in nanowire conductivity when analytes interact with their surface, due to the high nanowire surface-to-volume ratio. This new class of sensors will address future needs of Homeland Security for ultrasensitive detection, and our solution based fabrication will allow for simple and inexpensive production. Synthetically, we will grow nanowires of different compositions and will functionalize them with various organic functional groups in order to tailor their response to specific agents. On the fabrication front, we will use advanced lithography combined with alternating current (AC) dielectrophoresis to place nanowires between electrodes and perform electrical measurements on these devices. We completed proof-of-principle experiments by fabricating carbon nanotube devices using this method and showed that they have high sensitivity to chemicals. Our experimental efforts will be coupled with our first-principles modeling capabilities in quantum electronic transport. Such modeling will be necessary to understand the sensors to be built and to study the sensitivity of the nanowire conductivity to chemical, biological, and optical stimuli.

## Accomplishments

Our first goals this year were to fabricate initial nanowire devices and to understand their conductance properties without analytes. To this end, we developed a solution based nanodevice assembly method and fabricated devices based on carbon nanotubes. We developed a solution preparation method using single-stranded deoxyribonucleic acid as a surfactant. This allows us to dramatically improve the cleanliness and yield of our devices. Since contacts play a major role in nanodevices, we explored the effects of the type of metal (Pd, Au) on the device properties and developed a process to fabricate ultrathin oxide layers (PdOs) for electrical insulation of carbon nanotubes. We modeled the properties of these devices using different approaches. We developed a self-consistent, non-equilibrium, quantum transport approach to calculate the current-voltage characteristics of carbon nanotube devices. This was used to explain the robustness of our fabricated nanotube devices to conformational defects and to predict unusual short-channel effects. We also applied a tunneling theory to understand the properties of our nanotube/PdO contacts.

A second goal was to develop synthesis methods for new nanowires. We were successful in using solution based methods to grow ZnO nanowires that are much longer and narrower than previously achieved, and we also synthesized new Au nanoparticles that enhance the signals in surface enhanced raman scattering. In addition, we used vapor phase growth to synthesize GaN nanowires and GaN/InN core/shell structures.

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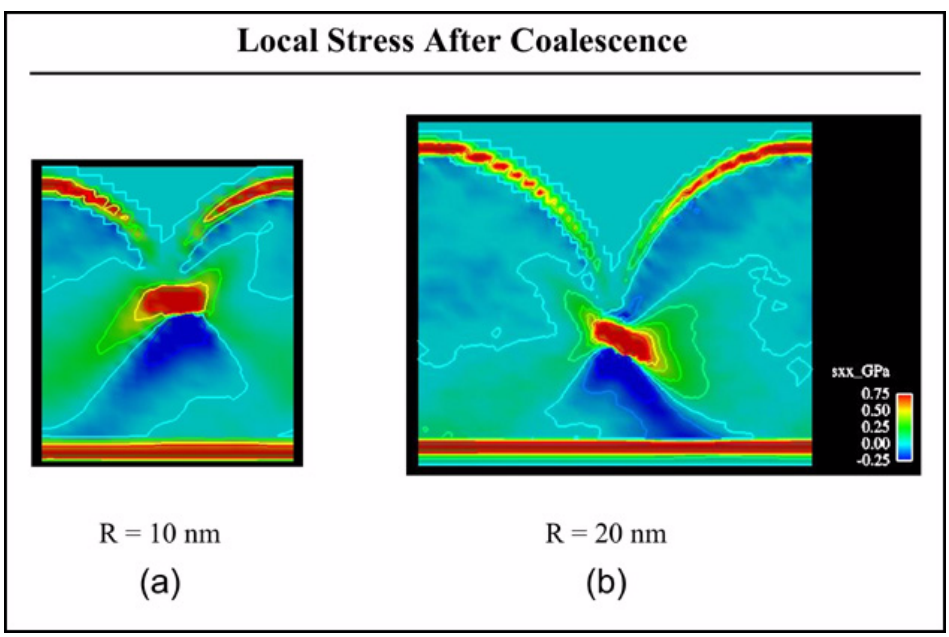
# Fundamental Enabling Issues in Nanotechnology: Stress at the Atomic Level

*79827*

*E. B. Webb III, J. J. Hoyt, S. M. Foiles, S. C. Seel, J. A. Floro, J. A. Zimmerman, S. J. Hearne, A. M. Morales*

## Abstract

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 strongly influence component lifetime and performance. Stress has also been proposed as a mechanism for stabilizing supported nanoscale structures. However, intrinsic connections between the evolving morphology of supported nanostructures and stress generation are still a matter of debate. We propose numerical simulations to predict stress generation mechanisms and magnitudes during all growth stages, from island nucleation to coalescence and film thickening. Simulations will be validated by electrodeposition growth experiments on patterned substrates, which can establish the dependence of microstructure and growth stresses on process conditions and deposition geometry. Deposition on nanopatterned substrates will permit an attempt to measure stress for isolated nanoislands. We will combine experiment and simulation to elucidate the role of interstitial adatoms in stress evolution during growth. Fully atomistic simulations will explore surface stress induced pressure (Laplace pressure) for isolated nanoislands as well as adatom behavior near islands. Island coalescence stress for island radii up to 0.25 micron will be studied with atomic scale detail; results will reveal structural changes associated with growth stress as a function of deposition conditions. A coupled atomistic/continuum method will expand simulation length scales permitting investigation of stress evolution during later growth stages, e.g., adatom incorporation into grain boundaries. Predictive capabilities of simulations permit direct determination of fundamental processes active in stress generation at the nanometer scale while connecting those processes to continuum models for much larger island and film structures. Coupling models with experiment permits validation and allows for deeper insight into existing data from in situ stress measurement experiments. Our results will reveal the materials science necessary to tailor stress and, therefore, performance in nanostructures and eventually in integrated nanocomponents.




*Stress distributions after coalescence for* (a) *R = 10 nm and* (b) *R =20 nm, calculated using our newly optimized homogenization technique. Note that a tensile stress band extends laterally throughout the R = 10 nm coalesced islands.*

## Accomplishments

We completed the first all-atom simulations of island coalescence using a system in which the adhesion energy between island and substrate was relatively high. To study stress before and after coalescence, we adapted a coarse-scale interaction homogenization technique for calculating stress in atomic simulations and optimized it to study isolated and coalesced island geometries. Our results demonstrated that coalescence stress increases for smaller islands in qualitative agreement with theory and experiment; however, the high adhesion condition simulated resulted in significant stress localization upon coalescence. This led to inelastic coalescence phenomena (void formation) for islands with radii 10 nm and smaller. While relevant to a class of growth conditions, the high adhesion case simulated does not accurately represent our experimental conditions used to measure coalescence stress because, in those experiments, islands are grown on glass or photoresist. As such, adhesion between the islands and substrate in the experimental setup is small, so simulations must be adapted to model this condition. In response, we developed a method for performing simulations of isolated islands and coalescence for island/substrate combinations with arbitrary adhesion. We also developed the first analytical model of island coalescence to explicitly consider the interplay between thermodynamics and mechanistics of coalescence. In collaboration with Northwestern University, we simulated neighboring Ge quantum dots on a Si substrate and fit a model to describe the variation of the interaction between islands as a function of island separation distance. Such knowledge is important to be able to predict stable nanoisland distributions resulting from strain-induced assembly.

In collaboration with Brown University, we investigated coalescence stress during patterned Cu film growth. We studied this system in response to prior work on Ni films where the film roughness presented complications to data interpretation. Roughness for Cu films was demonstrated to be two orders of magnitude less than for Ni; however, a number of problems arose (deposition bath stability, absence of clear coalescence signal in current). Therefore, we returned to studying Ni films. By studying coalescence stress on patterned surfaces with differing feature sizes, we hypothesize that roughness issues will be mitigated and a distinction will be established between stress generated by initial coalescence and continual coalescence after initial impingement. These experiments will directly address existing questions regarding the relative magnitude of various contributions to overall stress. We identified a novel patterning capability that promises patterned surfaces with finer features than have been used in prior coalescence stress studies. This will abet more detailed understanding of isolated island stress and coalescence stress for nanometer scale islands. Our initial results indicate that samples are not as monodisperse as desired; methods to improve this are under consideration.

## Other Communications

S.C. Seel, E.B. Webb III, and J.A. Zimmerman, “Stress Generation During Thin Film Growth: Island Coalescence Simulations,” presented at the 8th US National Congress on Computational Mechanics, Austin, TX, July 2005.

E.B. Webb III, S.C. Seel, and J.A. Zimmerman, “Atomic Scale and Continuum Simulations of Island Coalescence During Thin Film Growth,” presented at the Materials Research Society Annual Spring Meeting, San Francisco, CA, March 2005.

E.B. Webb III, S.C. Seel, and J.A. Zimmerman, “Atomistic and Continuum Simulations of Island Coalescence” presented at the American Physical Society Annual March Meeting, Los Angeles, CA, March 2005.

E.B. Webb III, S.C. Seel, J.A. Zimmerman, and J.J. Hoyt, “Island Coalescence Stress from Atomistic Simulations,” presented at the University of Wyoming Computational Mechanics Workshop, Laramie, WY, August 2005.

# Effective Dispersion of Nanoparticles by Polymers

*84266*

*D. L. Huber, A. L. Frischknecht, N. S. Bell*

## Abstract

Polymeric materials filled with dispersed nanoparticles show great promise for lowering the thermal expansion coefficient in nuclear weapon components and for use as high resolution photoresists for lithography of novel microsystems. The dispersion of nanoparticles is essential for a variety of other technologies important to Sandia, including high dielectric constant composites, ceramics processing, and solid state lighting. However, materials processing is a critical issue: stable nanoparticle dispersions are difficult to achieve because 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 will investigate the steric stabilization of nanoparticles by macromolecules. We will synthesize nanoparticles and attach polymers by functionalization or by polymerization directly off the particle surfaces. We will explore the effectiveness of various surface coverages, molecular weights, and charges for dispersing nanoparticles. The experiments and characterization will provide input for, and be interpreted using results from, self-consistent field and classical density-functional theory. Theoretical work will include predicting the structure of the polymeric layers and the interaction energy between coated nanoparticles. The ultimate goal of this project is to enable the use of dispersed nanoparticles in nanocomposite materials suitable for applications in nuclear weapon components and microsystems as well as other applications such as solid state lighting and ceramics.

## Accomplishments

We designed an entirely new method for synthesizing titania nanoparticles that is a convenient benchtop procedure, produces large quantities of nanoparticles, and uses inexpensive starting materials. These materials are already superior to the best commercial materials available, and are small enough to produce refractive index gains without light scattering. We also completed preliminary work on growing polymers from the surfaces of gold nanoparticles. We measured our ability to disperse a commercial titania nanopowder using steric stabilization and explored methods of improving the dispersions. We measured the zeta potentials of these materials and began creating dispersions of these materials in silicones. We also developed molecular dynamics simulations that clearly demonstrate the tendency of nanoparticles to agglomerate in polymer melts. These same simulations indicate that very small nanoparticles lack this tendency to agglomerate (assuming there are no energetically favorable interactions between the nanoparticles). We also set up some atomistic simulations of the titania steric stabilization systems.

## Other Communications

A.L. Frischknecht, R.S. Krishnan, A. Tuteja, M.A. Holmes, and M.E. Mackay, “Nanoparticle Surface Segregation in Polymer Thin Films,” presented at the Materials Research Society Meeting, Boston, MA, December 2004.

A.L. Frischknecht, R.S. Krishnan, A. Tuteja, M.A. Holmes, and M.E. Mackay, “Bulk and Interfacial Behavior of Nanoparticle/Polymer Blends,” presented at the American Physical Society Meeting, Los Angeles, CA, March 2005.

# Pulsed Power Sciences and High Energy Density Sciences

## Laser Triggering of Water Switches in Terawatt Class Pulsed Power Accelerators

*52738*

*J. R. Woodworth, G. S. Sarkisov, D. L. Johnson*

### Abstract

We propose to determine the optimum parameters needed to reliably laser trigger water switches in terawatt class pulsed power accelerators. Presently, water switches operate in a self-break mode, with the associated jitter in time and amplitude. The large jitter of self-breaking water switches forces the use of gas switches as master timing elements in multimodule accelerators. These gas switches are expensive, fragile, and difficult to make work at all above 5 MV. Replacing these gas switches with low jitter, triggered water switches will allow simpler, more robust accelerator designs that operate with higher field stresses and provide more energy to the load. In addition, triggering water switches in the ZR accelerator would allow improved timing between the power pulse and the Petawatt laser, allowing the Petawatt to be used in fast ignition fusion experiments. Laser triggering will initiate the streamer formation conditions at the electrode surface, provide an ionized path that behaves as an extension of the electrode, and provide trigger control and timing.

### Accomplishments

Previously, we demonstrated that green pulsed lasers could be used to trigger a 170 kV water switch with a command jitter of about 2 ns. Based on the 170 kV results, we designed and constructed a new pulsed power facility capable of testing laser triggered water switching at voltages ranging from 0.6 MV to 2 MV. We used two green lasers with pulse lengths of ~7 ns in conjunction with this facility. We used a 400 millijoule laser to trigger the switch and an ~100 millijoule laser as a back lighting source for a time resolved shadowgraphy system. The shadowgraphy system took pictures of the switch region while the water switch was in the process of breaking down.

We demonstrated that by focusing the laser beam through a hole in the cathode, we could form linear chains of point plasmas in the water gap between the switch electrodes using several lens systems, including spherical lenses, bifocal lenses, and axicon lenses. We used this technique to successfully trigger the 170 kV water switch. The axicon lenses gave the best chains of point plasmas for our use. These point-plasma chains rapidly expanded into a chain of neutral gas bubbles bridging part of the distance between the electrodes.

While we were able to command trigger the 0.6 MV water switch with the laser beam focused through the axicon lens, our results were marginal and suggest that much higher laser energies would be needed to command trigger 2 to 4 MV switches with low jitter. Since these larger lasers would have a cost comparable to the pulsed power facilities they were designed to trigger, the concept of laser triggered water switching

does not appear to be practical at the present.

## Refereed

G.S. Sarkisov, N.D. Zameroski, and J.R. Woodworth, "Observation of Electric Field Enhancement in a Water Streamer Using the Electro-Optical Kerr Effect," submitted to *Journal of Applied Physics.*

G.S. Sarkisov and J.R. Woodworth, "Measurement of the Current in a Water Discharge Using the Magneto-Optical Faraday Effect," submitted to *Journal of Applied Physics.*

J.R. Woodworth, D.L. Chalenski, G.S. Sarkisov, and J.R. Blickem, "170-kV Laser-Triggered Water Switch Experiments," to be published in *IEEE Transactions on Plasma Science.*

# Experimental and Computational Study of Liquid-Solid Transition in Tin

*52739*

*J. Davis, S. M. Foiles*

## Abstract

The behavior of materials under dynamic high pressure conditions is directly relevant to stockpile stewardship and is of scientific interest. In particular, there is a need for improved understanding of dynamic solidification behavior under high pressures. The purpose of this project is to study the solidification transition in tin under isentropic compression.

In FY 2005, we completed molecular dynamics simulations of solidification in tin using the previously developed interatomic potential, and determined the solid-liquid interface velocity over a range of pressures and temperatures. We made significant improvements in the experiment design and performed two experiments, but due to technical issues, neither of these provided high quality data.

The goal of this work was to develop future capability and expertise in a novel area of materials dynamics characterization that is of high interest to stockpile stewardship. The results may also improve the fundamental understanding of solidification processes, aiding both the prediction of material behavior under dynamic loading and the modeling of more traditional processes such as welding and soldering.

## Accomplishments

We conducted molecular dynamics simulations of liquid-solid interfaces to determine interfacial velocity as a function of temperature and pressure. We considered only the (001) solid crystal orientation. These simulations used the interatomic interaction potential developed during prior years of the project. The results demonstrate that the interfacial velocity varies linearly with the free energy difference between the solid and liquid phases, independent of whether the driving force is undercooling or overpressurization. We also used molecular dynamics simulations to determine the maximum superheating of the solid phase; this can be used to estimate the maximum undercooling and/or overpressurization of the liquid phase. (Isentropic compression involves overpressurization.) The simulations also provide an estimate of the solid-liquid interfacial free energy between the melt and the high pressure body-centered tetragonal phase of tin.

We executed two experiments on liquid tin at the Z Accelerator. We successfully addressed many of the technical issues, but the new experiments still failed to provide high quality data. This was due in part to problems with the facility's interferometer diagnostics. The main issue remaining to be addressed is the integrity of bonded interfaces under heating.

# Efficient Implicit Multigroup Radiation Calculations

*67120*
*T. A. Brunner*

## Abstract

Radiation transport is an important part of many high energy density physics (HEDP) simulations. Compared with other fields of physics, the short timescales of radiation transport require robust, fully implicit methods. Since the radiation transport calculations typically dominate the total run time of a solution and limit the time step, it is critical that the transport methods in the code run as efficiently as possible with a time step that is as large as possible.

Our radiation diffusion package now has a large and complete verification suite. This suite is managed with a new tool, Tampa, which is currently being adopted by the rest of the ALEGRA team for their own verification.

We developed two new preconditioners for the multigroup radiation diffusion package, each applicable in a different regime. With the appropriate new preconditioner, the solution of the coupled equations is more robust and accurate than with the old method.

The implicit Riemann solver work at the University of Michigan is advancing. They are developing a robust solver for the spherical harmonics equations that is potentially useful for our future problem sets.

## Accomplishments

We were successful in advancing the radiation simulation capabilities of the code, addressing the following milestones outlined for the year:

- *Milestone 1:* We developed an automatic verification for the radiation diffusion package. The suite contains 21 different test problems that are run on several different meshes resulting in nearly 700 simulations in the suite. Convergence studies are done in both two and three dimensions, and on regular, skewed, and randomized grids. As a result of this testing, we found many bugs in the radiation code, the NEVADA framework, and the linear solver packages in Trilinos.

  In order to manage this suite of problems, we developed a tool called Tampa. It manages the database of problems, combines them with the meshes (or other varying parameters), runs each of the simulations, and post-processes the results. The other physics developers in ALEGRA are adopting this tool, and other researchers are expressing interest.

- *Milestone 2:* We developed two new preconditioners for the multigroup radiation package. The first is a block diagonal preconditioner, where each of the diffusion equations for a group is solved independently of the others. This proved to be very effective for some of our problems, especially in cases with very little coupling of the radiation to the material and no rapid change. This essentially implements the old solution technique within a more robust method. The second preconditioner solves a one group problem in order to estimate the coupling between each of the groups, then, using this coupling, solves each group independently for the detailed energy spectrum. This should be fairly effective for more problems and larger time steps, but we have not yet performed exhaustive testing.

- *Milestone 3:* We did not yet examine or improve the time step control algorithm. The current method only allows a certain relative change in the radiation field from time step to time step. This may be sufficient for the new formulation of the diffusion solver, but the default maximum allowable change should probably be increased.

- *Milestone 4:* Our collaborators at the University of Michigan implemented an implicit Riemann solver for the spherical harmonics equations. They also modified the standard Riemann solver to preserve several important limits for the transport equation. This allows very large computational zones to be used in very diffusive regimes. They also started work on a multidimensional simulation capability.

## Refereed

T.A. Brunner, T.J. Urbatsch, T.M. Evans, and N.A. Gentile, “Comparison of Four Parallel Algorithms for Domain Decomposed Implicit Monte Carlo,” to be published in *Journal of Computational Physics.*

R. McClarren, J.P. Holloway, and T.A. Brunner, “Establishing an Asymptotic Diffusion Limit for Riemann Solvers on the Time-Dependent Pn Equations,” presented an in *Proceedings of M&C 2005 Avigion International Topical Meeting*, Avignon, France, September 2005, CD-ROM.

R. McClarren, J.P. Holloway, T.A. Brunner, and T.A. Mehlhorn, “An Implicit Riemann Solver for the Time-Dependent Pn Equations,” presented and in *Proceedings of M&C 2005 Avignon International Topical Meeting,* Avignon, France, September 2005, CD-ROM.

# Model for Channel Resistance in Water Breakdown

*67121*

*L. K. Warne, J. M. Lehr, R. E. Jorgenson*

## Abstract

Water breakdown switches are an integral part of the pulse compression schemes used in pulsed power machines. We determined the electrical properties of the switch from breakdown channel radius, length, and conductivity.

A physics model for water breakdown, based on a gas breakdown model, tends to overestimate channel resistance; as a result, researchers often use empirical substitutes. The gas breakdown model postulates that the channel conductivity, which is an input parameter, remains constant and the channel resistance varies over time because of shock expansion of the channel. There are alternative models based on a postulated constant channel radius (again an empirical input to the model) and a time varying conductivity as a result of channel heating.

Due to the large density of water that impedes channel expansion in the water breakdown model, the resulting increase in channel temperature and consequent increase in channel conductivity must be taken into account. This project is focused on incorporating the changes in channel conductivity with temperature into the model. This both improves the correlation with experimental results and eliminates the need for empirical conductivity input. An accurate water breakdown switch model based on physical principles will allow us to conduct simulations for the design of future higher amplitude pulsed power machines.

## Accomplishments

We ran and studied many one-dimensional simulations to examine the characteristics of the water channel expansion and heating. We focused on the later time expansion phase of the water breakdown switch, and hence assumptions are still made regarding initial conditions of the channel. Our simulations used initial channel radii in the 1 to 10 micron range. Temperatures of 25 to 70 electron volts were reached during the subsequent late time phase, which led to electrical conductivities that were an order of magnitude larger than those used in the previous water breakdown model. Water conductivities grow somewhat faster with temperature than atmospheric gases, and thus it is important to capture the effect of these temperature increases in the model.

*Water switch experiment.*

Examination of channel opacities indicate that open radiation, as assumed by the gas breakdown model for gas channels, does not accurately apply here. As the channel expands, the optical thickness results in an eventual black body emission.

Improvements in a zero-dimensional lumped model for the conductivity of the channel versus temperature, the ionization number, and radiation resulted in good agreement with the one-dimensional simulations. We made all of these improvements to more accurately represent the underlying physical phenomena (not fitting the final switch resistance).

We made some further simplifications to extract a model similar to the water breakdown model, but with temperature and conductivities determined in the solution process that can be implemented in circuit codes.

Finally, we performed comparisons to experiments on Z20 against the models. We used circuit tools to extract reflections from the data to allow resistive fall levels to be accurately estimated.

FY 2005 accomplishments:

(1) We created one-dimensional ALEGRA simulations for slow and fast rise time current drive circuits as

well as voltage drive circuits.

(2) We improved the zero-dimensional model conductivity function, ionization level, and radiation.

(3) We extracted a simple circuit model of similar character to the previous water breakdown model, with temperature and conductivity determined as part of the solution.

(4) We compared experimental data from Z20 using circuit extraction of reflections to obtain improved resistive fall levels for the water switches.

# Embeddable Shock Physics Sensors

*67122*

*T. A. Friedmann, J. Mason, M. D. Knudson, R. E. Setchell, T. J. Vogler, J. Davis*

## Abstract

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, methods must be developed to embed small, non-perturbing 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. We expect that data generated from these sensors will allow 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.

## Accomplishments

This year, we focused on building up aluminum targets for gas gun experiments. Using pulsed laser deposition, we fabricated thin film thermocouple gauges for temperature measurement and thin film manganin gauges for stress measurements on top of dielectric layers of either $Al_2O_3$ or $SiO_2$. Problems with defects in the dielectric layer led to shorting of the gauges, and much more work remains to overcome this problem. We are switching to nonconducting sapphire or fused silica targets in order to prepare samples for gas gun experiments. We performed finite element modeling of the temperature response of the thin film thermocouple gauges heated by laser pulses. The calculations and the measured temperature response are in reasonable agreement, indicating that it should be possible to extract accurate temperature data from dynamic measurements. We procured equipment for measuring the response of electrical gauges on Z in order to look at the noise response of commercial thermocouples in Z.

# Characterizing the Emissivity of Materials Under Dynamic Compression

*79877*

*D. H. Dolan III, J. Podsednik, J. Davis*

## Abstract

Pyrometry is an important part of dynamic material studies; it provides one of the only means of measuring temperature in shocked and isentropically compressed samples. Temperature measurements are necessary for the development of complete equations of state for compressed materials but are often ambiguous because the analysis requires knowledge of a sample's emissivity, or deviation from ideal blackbody behavior. To deal with this problem, we developed a high emissivity thin film coating for use in dynamic temperature measurements. By characterizing the behavior of this coating as a function of temperature and pressure, we hope to create an emissivity standard that can be used in a broad range of dynamic compression experiments.

We focused on three types of characterization for the high emissivity film in FY 2005. First, we analyzed the composition and structure of the films to better understand why the films have such a high emissivity and to identify potential shortcomings of the films in dynamic compression experiments. Next, we performed static emissivity characterization of the films at ambient conditions and elevated temperatures in collaboration with the National Institute for Standards and Technology (NIST). Finally, we developed a method for probing the emissivity of the coatings under shock wave compression. We will perform dynamic emissivity measurements by coupling a Sandia impact system to an infrared beam line of the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory.

## Accomplishments

Most of our effort focused on a chromium based film developed at Sandia, which thus far proves to provide an effective and robust way to enhance the emissivity of various materials in the infrared spectrum. Transmission electron microscopy studies verify that the films consist of a two layer metal/metal oxide with nanoscale porosity; x-ray photoelectron spectroscopy confirms that the second layer of the film is largely composed of various chromium oxides. We also launched additional work on rhenium silicide to complement the chromium film effort.

We performed static emissivity measurements of the chromium based films at Sandia and NIST. At ambient conditions, these measurements are very consistent, affirming the high emissivity of our films. With Sandia support, NIST is developing systems for measuring the emissivity of thin films at elevated temperatures (600–1300 K).

Preparations for dynamic emissivity measurements focused on creation of a small impact system. We are currently testing a prototype of the system, and construction of the final system will begin shortly. We are preparing to bring the impact system to the synchrotron at Brookhaven National Laboratory in FY 2006. We conducted a preliminary evaluation of the technical feasibility; and further arrangements regarding safety, floor space, beam time, and interim storage are in progress. Work is also under way to identify time resolved infrared diagnostics capable of detecting individual light pulses from the synchrotron.

We used preliminary infrared imaging experiments to demonstrate that chromium coatings retain their emissivity enhancement at stresses above 10 GPa.

# Beyond the Local Density Approximation: Improving Density Functional Theory for High Energy Density Physics Applications

*79878*

*M. P. Desjarlais, M. P. Sears, R. P. Muller, A. E. Mattsson, A. F. Wright, N. A. Modine*

## Abstract

Density Functional Theory (DFT) has become the tool of choice for developing manifestly consistent equations of state, electrical conductivities, and opacities for materials of interest to Sandia's high energy density science (HEDS) programs, particularly in the difficult warm dense matter regime. While DFT is a formally exact reformulation of the Schroedinger equation, the true form of the exchange correlation functional (Exc) is unknown. While currently available functionals provide sufficient accuracy for many applications in physics and chemistry, they also exhibit highly consequential errors, such as an underestimation of the bandgaps in insulators and semiconductors. At lower temperatures, this can lead to incorrect calculations of the opacity and electrical conductivity. In HEDS applications, we have the added consequence that excited states lying above the incorrectly computed gap are thermally occupied, resulting in errors in the energy and pressure. In deuterium, the largest differences between DFT results and Sandia data occur in the regime where improvements in the DFT bandgap would be most significant. The exchange part of the Exc functional can be constructed exactly, and doing so greatly improves bandgap calculations. Including exact exchange in DFT is currently of great interest. We fully expect that improvements to the bandgap calculations will find broad use outside the HEDS applications. We implemented exact exchange (EXX) in Sandia's plane-wave density functional code Socorro and are beginning to test it on HEDS and materials science applications. We began initial work on developing an appropriate correlation functional compatible with EXX. We generated compatible EXX pseudopotentials for use in the calculations. We also achieved considerable optimization of the EXX algorithms.

## Accomplishments

Our EXX project is structured around three major tasks: Socorro code development, EXX pseudopotential generation, and correlation functional development. We made good progress in all three areas.

We implemented an initial EXX capability in the Socorro electronic structure code. This required implementation of two primary components, an iterative optimized effective potential (OEP) approach and the calculation of exchange integrals. In addition, we made considerable progress toward FY 2006 milestones requiring optimization and refinement of the EXX implementation.

The central algorithm in our iterative OEP approach calculates the gradient of the energy functional with respect to the local potential appearing in the Kohn-Sham equations. Since the energy functional depends implicitly on the local potential via the solutions of the Kohn-Sham equations, the calculation of this gradient is not straightforward. Working with Dr. Ross Lippert from the Mathematics Department at the Massachusetts Institute of Technology, we found a new derivation of the required gradient based on the density matrix. We implemented the gradient expression (with finite temperature terms) in the Socorro code. In order to perform standard DFT calculations within the local density approximation (LDA), we incorporated the results into a minimization algorithm. With this technique, we verified the correctness of our new approach.

We derived detailed expressions for the exchange integral and the derivative of the exchange integral with respect to a Kohn-Sham function. We integrated the divergences that appear in these expressions using a scheme published in the literature. We parallelized the new subroutines added to Socorro to perform this integration, but did not yet optimize their performance. The published scheme uses the addition and subtraction of a certain function to convert the exchange energy into a non-singular integral, which we can evaluate by sampling. The function given in the literature is specified only for face-centered cubic crystals and is not applicable to general unit cells. We extended the scheme by developing a function that is applicable to any unit cell and that has all the desired properties.

We coupled our iterative OEP algorithm with our implementation of the EXX integrals to get an initial EXX capability in the Socorro code. We tested our implementation by calculating the bandgap of bulk sili-

con. Using a 3x3x3 Monkhorst-Pack sampling of the Brillouin zone of the two atom unit cell, we calculated a 1.22 eV indirect bandgap for silicon. This can be compared with an indirect bandgap of 1.17 eV from the experiment and 0.47 from the LDA, demonstrating the improvement in calculated bandgaps that we expect from the EXX.

We developed an auxiliary all electron capability for EXX calculations on small systems. Our code currently reproduces published numbers for $H_2$ and He. This all electron capability gives us the ability to benchmark the EXX results against Hartree-Fock and other wave function based results, and will assist in generating EXX based pseudopotentials. We are currently using the FHI98PP software to derive pseudopotentials based on the simplification of EXX due to Krieger, Li, and Iafrate (KLI), which reproduces EXX results for many systems.

We are using the previously published “subsystem functional” scheme to construct a partially compatible EXX correlation functional. We are testing a preliminary version. We expect our EXX correlation functional to do well in solid state systems with large density variations, such as bulk silicon. It may need modification to be able to correctly reproduce, for example, finite size effects. Based on our test results, we will modify the functional to improve the performance.

# Exploring Pulse Shaping for Z Using Graded-Density Impactors on Gas Guns

*79879*

*M. D. Furnish, J. Davis, W. D. Reinhart, M. E. Kipp*


## Abstract

While isentropic compression experiment (ICE) techniques have proven useful in deducing the high pressure compressibility of a wide range of materials, they have encountered difficulties where large volume phase transitions exist. We sought to apply graded-density impactor methods for producing isentropic loading to planar impact experiments to such selected problems. We chose cerium due to its 20% compression between 0.7 and 1.0 GPa. We constructed a model based on limited earlier dynamic data and applied it to the design of a suite of experiments. We installed a capability for handling this material. We executed two experiments using shock/reload techniques with available samples, loading initially to near the gamma-alpha transition, then reloading. We also conducted two graded-density impactor experiments with alumina. We developed and validated a method for interpreting ICE data that uses a wavelet construction for the ramp wave and includes corrections for the "diffraction" of wavelets by releases or reloads reflected from the sample/window interface. We assessed alternate methods for constructing graded-density impactors.


## Accomplishments

We chose cerium metal, an outstanding example of a material with a large volume change phase transition (strain of 20% at the gamma-alpha transition at 0.7–1.0 GPa), as a sample material for ICE concept validation. A model described in the literature could not be readily implemented into the CTH code to be used for our analyses. However, we used the principles of the published approach to formulate parameters for a current phase transition model existing within the code: the "PTRAN" model. We validated this code with the single shock data, Hugoniot data, and static data in the literature.

We designed a set of experiments to exercise multishock and ICE compression of cerium, making use of the CTH numerical models described above. We executed two multisample experiments using shock-reload techniques and velocity interferometer system for any reflector (VISAR) diagnostics, loading initially to just below or just above the gamma-alpha transition, then reloading. Marked relaxations of the sample velocities between the loading and reloading suggest that the phase transition may be affected during this period for both shots. The plateau amplitudes differ by about 25% (0.67 GPa and 0.85 GPa).

We conducted two graded-density impactor experiments with alumina (AD995) samples, utilizing 7-layer graded-density impactors. The data from these tests provided continuous high quality compression curves for the alumina, as well as validating a multilayer construction of a "pillow impactor."

We developed a method for interpreting ICE data using a "wavelet" construction for the ramp waves. It includes corrections for the "diffraction" of wavelets by releases or reloads reflected from the sample/window interface (a substantial bookkeeping task). It includes both Hugoniot and isentropic representations of the optical windows. We validated the method with several tests: a molybdenum test to 50 GPa with LiF windows (providing a substantial impedance mismatch between the sample material and the windows), an aluminum test with LiF windows, and the alumina test mentioned above.

# Thermo-Physical Properties of Shocked Water for Modeling Pulsed Power Switches and Other HEDP Systems

*79880*
*T. K. Mattsson, L. K. Warne, M. P. Desjarlais*

## Abstract

Water is used in breakdown switches and in tampers in exploding wire experiments. Since predictive modeling depends crucially on high fidelity models of thermo-physical properties, it is important to know these properties for water. In this project, we develop an equation of state (EOS) model, including electrical conductivity (sigma), for water. The difficult regime with temperatures between 0.5 eV and 3 eV (5000 K to 30,000 K), the so-called warm-dense-matter (WDM) regime, is of particular interest. We employ state-of-the-art simulation techniques, quantum molecular dynamics (QMD) to model the system from first principles. Significant development of the current approach is necessary in order to treat a molecular liquid where the chemical equilibrium changes with temperature and pressure. The dual nature of conductivity in water is an added complexity compared with most previously studied systems and requires substantial development. The resulting QMD values for EOS and sigma are directly applied to validate and improve existing circuit models for plasma channels in water breakdown switches. Furthermore, we will use the QMD results to create tables for use in radiation-hydrodynamics ("rad-hydro") codes, e.g., ALEGRA, thus improving the fidelity of macroscopic modeling efforts.

## Accomplishments

We met all FY 2005 milestones: to develop a chemical composition model and select Density Functional Theory (DFT) simulation parameters, to select an adequate EOS, and to gain a better understanding of plasma channel conditions.

We made significant progress in determining parameters for DFT simulations and developing post-processing tools in Mathematica for multicomponent systems. We are able to calculate chemical composition, pair correlation functions between different species, and diffusion coefficients for both hydrogen and oxygen. The diffusion is calculated using the mean displacement method, as well as the Fourier transform of the velocity-velocity correlation function.

In parallel, we investigated the conditions in plasma channels using rad-hydro simulations. With ALEGRA, we found that during the initial 10 ns of breakdown, water is taken from standard conditions of 300 K and 1 g/cm$^3$ to between 2.5 g/cm$^3$ and 3000 K, to even less than 0.1 g/cm$^3$ and 300,000 K.

With the QMD EOS results benchmarked with the state-of-the-art EOS, where available, we are now confident in the method and will proceed to the regions of phase space now known to be of most interest to electrical breakdown in water switches. So far, we covered densities between 0.1 g/cm$^3$ and 3 g/cm$^3$ for temperatures between 2000 K and 70,000 K. We gained much new knowledge of conductivity in the region of phase space involved in the breakdown processes.

The QMD simulations validate the SESAME EOS for temperatures above 40,000 K and temperatures below 1000 K. In the intermediate regime between 2000 K and 20,000 K, we found that the SESAME EOS overestimates the dissociation of water and hence predicts a too high pressure. Since the deviations are in a relatively localized part of phase space, we plan to augment the SESAME table in that region. Although the corrections are in a localized region, this region is of importance to simulations of water breakdown switches. Hence, the adjustments are potentially important.

From a methodological standpoint, calculating the proton conductivity emerged as the most challenging problem. While the regime where there are many free protons can be straightforwardly analyzed with the Green-Kubo transport formalism, the transition from insulating to conducting is more difficult to describe. We developed a method to calculate in particular that transition to proton conduction more accurately than in the past. The calculated conductivity is in agreement with experimental data, where available.

Electrical conductivity of water appears at two branches in phase space: electronic conductivity dominates for all densities for temperatures above 8000 K, while ionic conductivity becomes important at 2000 K for densities above 2 g/cm$^3$. At 4000 K and 2.3 g/cm$^3$, there is a crossover from ionic to electronic conduction.

The conductivity isotherms below 8000 K show a markedly different behavior than isotherms above. The differences will most importantly affect electrical breakdown simulations in the shocked regions of phase space (2000–4000 K and 2–3 g/cm$^3$).

# Triggered Low Inductance Gas Switching

*79881*

*H. C. Harjes, M. A. Kincy, J. M. Elizondo-Decanini, K. W. Struve, J. P. Corley*

## Abstract

We are investigating methods for altering the configuration of a laser triggered gas switch in order to reduce its inductance. Reducing this inductance would improve the performance of a ZR type machine. If inductance can be reduced enough, it may be possible to eliminate a stage of pulse compression. We would achieve the lower inductance by increasing the diameter of switch electrodes (approaching the diameter of the intermediate store and pulse forming line) while maintaining multichannel arcing in the cascade section. We thoroughly investigated the switch concept with electrostatic computer codes to develop a demonstration switch design, and completed the mechanical design of the switch. We will construct and test it at the 1.5 MV level, at the University of Missouri (MU). We plan to use what we learn to begin the design of a full scale 6 MV switch.

## Accomplishments

In present pulsed power systems, the laser triggered gas switch (LTS) is typically positioned between the intermediate store (IS) and the pulse forming line (PFL). These lines are normally coaxial with inner conductors several (4–5) feet in diameter. When closed, the LTS electrically connects the inner conductors. The inductance of the switch, in large part, determines the energy transfer time from the IS to the PFL. The primary purpose of this project is to investigate and demonstrate methods for significantly reducing this transfer time.

The LTS consists of a trigger section that is closed by a single on axis arc and a cascade section that is closed by multiple arcs. The cascade section has a diameter of less than one foot. Since the inductance of a coaxial cable is proportional to the logarithm of the ratio of outer diameter to inner diameter, the small diameters at which the switch arcs occur make the switch more inductive than it may have to be. We are investigating alternative switch configurations in which the arcs are moved to diameters comparable to that of the IS and PFL. If we can maintain multichannel operation in these configurations, the switch inductance will be significantly reduced. In order to operate properly, the open state electric field distribution in the switch gaps must be carefully graded. We conducted electrostatic field simulations on alternative designs to determine if the proper field grading can be achieved. We investigated several alternative configurations from which we selected a configuration for demonstration testing. We completed a detailed electrical and mechanical design and completed preparations for testing. A summary of our accomplishments follows:

(1) We investigated three options for the electrode/cascade region configuration.

(a) Linear gap with nonlinear electrode diameters. This option allows a nontapered (cylindrical shell) outer ground shell, but the inductance is not as low as possible.

(b) Nonlinear gap with the same diameter electrodes on the cascade. This configuration can be implemented if the outer metal shell can be tapered somewhat; this configuration results in the lowest inductance possible.

(c) We incorporated the hybrid-trigger section to eliminate the trigger inductance penalty.

(2) We successfully developed a 1.5 MV demonstration switch design.

(a) Electrical and mechanical designs are complete.

(b) Drawings are nearing completion.

(c) We began ordering materials.

(3) We secured a test bed at MU for demonstration tests. MU personnel participated in the design of the demonstration switch to ensure compatibility with their test bed.

# Finite Temperature Self-Consistent GW Approach: Applications to High Energy Density Physics

*84272*
*S. V. Faleev, M. P. Desjarlais*

## Abstract

Reliable and accurate theoretical calculations of electronic structure, electrical conductivities, and opacities of materials at high temperature are very important for high energy density physics (HEDP). Results of these calculations are integral and crucial components of modern advanced simulation codes. Existing calculations of materials properties generally use the local density approximation (LDA) within density functional theory (DFT). This causes systematic errors in the electronic and atomic properties, in large part because the electron interacts with itself through the total electron density. In particular, the bandgaps of almost all materials are systematically underestimated in LDA. In HEDP applications, the LDA bandgap error affects the conduction electron population and, correspondingly, the pressure, conductivity, and opacity.

We hope to eliminate the self-interaction problem by computing the electron self-energy using a self-consistent implementation of what is commonly known as the self-consistent GW approximation (scGWA) for calculation of electronic structure. Sandia possesses one of the few implementations of scGWA in the world; this unique tool is very accurate and highly efficient. Most properties of weakly correlated systems calculated with the present zero temperature scGWA are much better than LDA predictions, particularly for insulators and semiconductors. We propose to extend the scGWA method to HEDP temperatures. Such an extension requires significant programming efforts, but the resulting new capabilities would constitute a major advance in ab initio methods for HEDP and would provide the world's first GW method suitable for high energy density physics.

## Accomplishments

We developed a new ab initio method for electronic structure calculations at finite temperature based on the all electron quasiparticle scGWA and Keldysh time-loop Green's function approach. We applied our method to five materials with a wide range of bandgaps (InSb, Ge, Si, GaAs, and diamond). We explored fundamental questions regarding how the bandgaps and quasiparticle (QP) energy levels vary with electronic temperature in the scGWA approach, and how this compares with the functional theory LDA (FT-LDA). In both approaches, we interpret the Kohn-Sham eigenvalues spectrum as an electron spectrum.

To the best of our knowledge, these are the first such FT-GW calculations of the bandgaps for semiconductors and insulators. We show that the GW bandgaps universally decrease with temperature, in contrast with the FT-LDA where the bandgaps universally increase. At temperatures of a few eV, the difference between the FT-GW and FT-LDA QP energy levels is significantly reduced compared to that at zero temperature. This is an important result, showing that existing high temperature calculations based on the FT-LDA are more justified than might appear from the known LDA bandgap errors at zero temperature. This result might serve as a justification of FT-LDA applicability to simulations of warm dense matter and as a useful foundation for correcting FT-LDA bandgaps. These results are of considerable importance for HEDP because it remains numerically very challenging to use approaches beyond FT-LDA for description of many atoms in large unit cells employed in HEDP simulations. Still, the GW calculations must achieve 0.1 eV accuracy for calculated QP levels.

# Science and Technology Strategic Objectives

## New Self-Assembled Nanocrystal Micelles for Biolabels and Biosensors

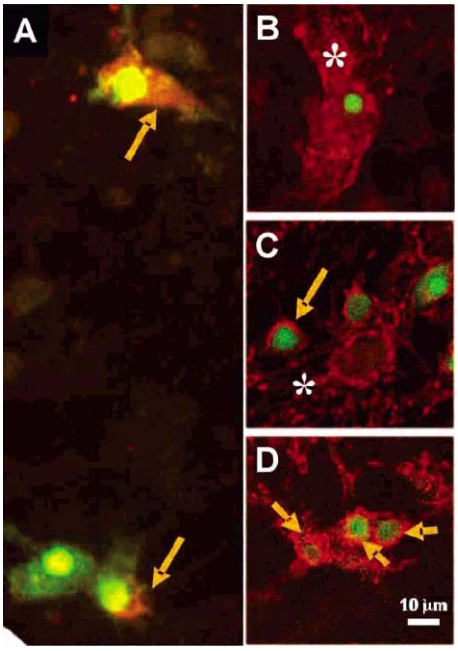
*69163*

*H. Fan, T. J. Boyle, C. J. Brinker*


### Abstract

The ability of semiconductor nanocrystals (NCs) to display multiple (size-specific) colors simultaneously during a single, long term excitation holds great promise for their use in fluorescent biolabeling. The main challenges of using NCs as biolabels are achieving biocompatibility and low non-specific adsorption, while avoiding aggregation. We propose a new, general method to directly mass-produce water-soluble NC-micelles that incorporate sites for bio-functionalization. Our approach is to encapsulate as-synthesized, hydrophobic NCs within the hydrophobic cores of water-soluble micelles composed of a mixture of nonionic polyethylene glycol (PEG) and amine terminated phospholipids. Success of this project will lead to unique capabilities at Sandia in producing specific protein tags for understanding biological behavior.


In FY 2004, we demonstrated a new route to the synthesis of water-soluble and biocompatible NCs. We accomplished this through the encapsulation of as-synthesized, monosized, hydrophobic metal and semiconducting NCs within the hydrophobic cores of micelles composed of a mixture of surfactants and phospholipids containing functional head groups such as polyethylene glycol (-PEG), -COOH, and $–NH_2$ groups, etc. In FY 2005, we turned our efforts to characterization and utilization of these water-soluble NC-micelles. We evaluated the biocompatibility of fluorescent NC-micelles by examining their uptake in cultured rat hippocampal neurons. While the route through which quantum dots (QDs) are taken up by cells has yet to be resolved fully, they do appear to accumulate in intracellular vesicular compartments in several cell types, suggesting uptake may be mediated, at least in part, though endocytosis. We extended the surfactant/lipid encapsulation techniques to synthesize water soluble magnetic NC-micelles. Transmission electron microscopy (TEM) results suggest that water-soluble magnetic NC-micelles exhibit no aggregation. The resulting NC-micelles preserve the magnetic properties of the origi-




*Images of CdSe/CdS micelles, prepared using modified phospholipids, in cultured rat hippocampal neurons.*

nal hydrophobic magnetic NCs. Viability studies, conducted using yeast cells, suggest that the magnetic NC-micelles are biocompatible. We demonstrated for the first time that by using external oscillating magnetic fields to manipulate the magnetic micelles, we can kill live cells, presenting a new magnetodynamic therapy without side effects.

## Accomplishments

We synthesized water-soluble fluorescent semiconducting NC-micelles. To evaluate the biocompatibility of these NC-micelles, we performed initial studies to examine their uptake in cultured rat hippocampal neurons. While the route through which QDs are taken up by cells has yet to be resolved fully, they accumulate in intracellular vesicular compartments in several cell types, suggesting uptake may be mediated, at least in part, through endocytosis. Because synaptic vesicle recycling is tightly coupled to neurotransmitter release, endocytosis can be manipulated. Depolarization of the plasma membrane excites neurons, evoking exocytosis for neurotransmitter release, followed by endocytotic retrieval of synaptic vesicle membrane from nerve terminals. QD-micelles accumulate in neurons and are distinguished by immmunohistochemical counterstain for NeuN after exposure for 16 hours in basal serum-free growth media. Three-dimensional (3-D) reconstruction of confocal images confirms the intracellular localization of QD fluorescence after long term exposure. That QDs retain their fluorescence character after lengthy incubation suggests that QDs can accumulate without aggregation and general compatibility of the micelle coating within a cellular environment.

We extended the surfactant/lipid encapsulation techniques to stabilize and self-assemble NCs with other shapes including rods and cubes. We prepared CdTe nanorods by using trioctylphosphine oxide as a stabilizing agent. We conducted encapsulation by using cetyltriemthylammonium bromide (CTAB). The TEM image revealed that surfactant “bilayer” stabilized CdTe nanorods formed uniform arrays. We prepared $Fe_2MnO_4$ nanocubes using oleylamine as a stabilizing agent. We conducted the formation of $Fe_2MnO_4$ nanocube-micelles using CTAB. The TEM image showed that the $Fe_2MnO_4$ NC-micelles self-organize into ordered arrays with cubic symmetry. Incorporation of magnetic particles in cells suggests other possible applications.

We demonstrated for the first time that by using external oscillating magnetic fields to manipulate the magnetic micelles, we can kill live cells. Preliminary data using yeast cells treated with biofunctionalized magnetic nanoparticles ($Fe_2MnO_4$) and oscillating magnetic fields (frequency between 12.5 to 100 kHz, magnitude 2.5 to 5 milliTesla, for 5 minutes) showed 80 to 99% cell killing, with no killing by MNP or fields alone. Our ultimate goal is to produce biocompatible magnetic nanoparticles, bioconjugating them with tumor specific targeting antibodies to demonstrate tumor killing efficacy.

We developed another new synthetic route to functionalize NCs with organosilane groups through encapsulation of monodisperse, hydrophobic gold NCs within the core of silane precursor micelles. Octadecyldimethyl(3-trimethoxysilylpropyl)ammonium chloride is a special functional silane with amphiphilic characteristics that can self-assemble into micelles and liquid crystal mesophase. After encapsulation, the micelles are monodisperse, exhibit hydrophilic characteristics, and can self-assemble into 3-D ordered arrays. In comparison with previous methods concerning the preparation of hybrid silica with stabilized gold NCs, this method is simple and general, allowing further grafting/functionalization of other functional groups through siloxane chemistry.

We observed the ultraviolet/visible (UV/vis) spectra of dodecanethiol (DT)-stabilized gold NCs in chloroform. The absorbance at ~519 nm corresponds to the gold surface plasmon resonance band. Through comparison, we observed no difference in the positions and peak widths of the plasmon resonance band of the DT-stabilized gold NCs in chloroform, the corresponding water-soluble NC-micelles, or the solid film of ordered arrays. We also observed no difference after addition of hydrogen chloride aqueous solution. This suggests that gold NC-micelles remain monodisperse in water and in solid films without aggregation. Additionally, the encapsulation process does not change the optical properties of DT-stabilized NCs. In TEM images, the ordered hexagonal arrays indicate no aggregation of the NC-micelles.

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# Superhydrophobic Surface Coatings for Microfluidics and MEMS

*73185*

*F. B. Van Swol, S. Singh, C. J. Brinker, J. E. Houston, T. L. Sounart*

## Abstract

Low solid interfacial energy and fractally rough surface topography confer to the Lotus plant superhydrophobic (SH) properties like high contact angles, rolling and bouncing of liquid droplets, and self-cleaning of particle contaminants. This project exploits the porous fractal structure of a novel synthetic SH surface for aerosol collection, its self-cleaning properties for particle concentration, and its slippery nature to enhance the performance of fluidic and microelectromechanical system (MEMS) devices. We aim to fundamentally understand the conditions needed to 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 micro-channel. 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 pre-defined/micromachined fluidic architecture. The fluid/solid boundary condition is important because it governs flow resistance and rheology and establishes the fluid velocity profile.

In our recent experiments, we studied fluid flow in the vicinity of textured SH coated surfaces using both rolling spheres on an inclined plane and a hydrofoil. The results indicate there is evidence of considerable drag reduction in the combined presence of texture and SH coating, causing droplets to roll faster. The highest velocities were reached on a textured surface with a characteristic size of ~8 micrometers. We used the Interfacial Force Microscope (IFM) and neutron reflectivity (NR) experiments to probe the equilibrium properties of the solid/fluid boundary. The IFM experiments show that when the gap between two submerged SH surfaces is less than ~1–2 micrometers, cavitation takes place, transforming liquid water to water vapor in the gap.

## Accomplishments

We continued our experimental, theoretical, and simulation studies of porous, superhydrophobic surfaces in contact with water. Our goal is to understand new phenomena like the rolling and bouncing of water drops, slip at the water/SH boundary, water exclusion from submerged SH surfaces, and cavitation between approaching submerged SH surfaces. In studying the flow of water on grooved SH surfaces, we showed, for the first time, effective macroscopic slip with slip lengths consistent with the characteristic sizes of the surface features and slip velocities exceeding 1 cm/s. Slip at the SH water interface was also probed in a series of multiresonant acoustic wave experiments. Our observation of very low attenuation for submerged SH surfaces is also consistent with a slip boundary condition. We conducted rheology experiments to provide an independent measurement of slip. We performed these experiments using the particle image velocimetry (PIV) system and a rheometer, using a custom built narrow-gap Couette setup that contains an SH coated, grooved surface on one side. The rheometer measures the torque and has been designed to overcome optical problems that may otherwise compromise PIV measurements. Preliminary results show an 8–10 % reduction in the torque, indicative of significant slip at the boundary.

We conducted NR experiments to probe the interface between water and a porous surface as the water contact angle was successively reduced from 160 to 100 to < 10 by photocalcination with ultraviolet (UV) exposure (here the UV exposure changes the surface chemistry while maintaining the original surface roughness). We showed that for submerged SH surfaces (contact angle = 160), water is completely excluded from the film interior and penetrates to a level (~10 nm) consistent with the scale of the surface roughness. We initiated NR studies to examine the corrosion protection potential of an SH coating applied to an aluminum surface. We found that after 15 hours of exposure to a $NaCl/D_2O$ solution, an unprotected sample exhibited significant changes in reflectivity fringes indicative of structural damage at the aluminum surface. In contrast, when we applied an SH coating, the reflectivity showed no changes even after nearly 200 hours of exposure. In addition, we are adding nanostructured $CeCl_3$/Silica particles to the SH film to increase roughness and increase contact angles and to provide corrosion inhibitors to the SH film.

Last year, we discovered that cavitation occurs between two submerged SH surfaces at a separation of 1–2 micrometers. This year, we performed additional experiments and simulations to understand this cavitation event. Molecular dynamics simulations of water confined between two plates with opposing SH patches captured many features of the cavitation event. Cavitation proceeds by the coalescence of fluctuating vapor films emanating from each of the opposing SH surfaces. IFM measurements show that cavitation corresponds to a highly non-linear attractive force of relevance to adhesion and natural phenomena like protein folding.

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# Adaptive-Responsive Nanostructures for Sensing Applications

*79882*

*J. A. Shelnutt, C. J. Medforth, F. B. Van Swol*

## Abstract

Functional organic nanostructures, such as well formed tubes or fibers that can easily be fabricated into electronic and photonic devices, are needed. Especially desired are such nanostructures that have enhanced sensitivity for the detection of chemical and biological (CB) agents and other environmental stimuli. We recently discovered a family of highly responsive and adaptive porphyrin nanostructures (tubes, rods, and more complex forms) that may satisfy these criteria. These novel porphyrin nanostructures, which are formed by ionic self-assembly of two oppositely charged porphyrins, may function as conductors, semiconductors, or photoconductors. They also have additional properties that make them suitable for device fabrication (e.g., as ultrasensitive colorimetric CB microsensors).

Preliminary studies show that these nanostructures have novel optical and electronic properties, including strong resonant light scattering, quenched or enhanced fluorescence, and electrical conductivity. Additionally, they are photochemically active and capable of light harvesting and photosynthesis, and they may also have nonlinear optical properties. The porphyrin nanotubes, for example, are mechanically responsive and adaptive, i.e., the rigidity of the micrometers-long nanotubes is altered by light, ultrasound, or chemicals; and the tubes are self-healing. Given the tremendous degree of structural variation possible in the porphyrin subunits, additional types of nanostructures and control over their morphology could be anticipated and has been realized. Molecular modification also provides a means of controlling their electronic, photonic, and other functional properties. We are establishing the range of ionic porphyrin nanostructures that can be made, and we are determining their electrical, optical, and responsivity properties. We are exploring the means for controlling their morphology, size, and placement on surfaces. We are also investigating their extraordinary photonic, electronic, and mechanical switching properties. This research lays the groundwork for the use of these remarkable porphyrin nanostructures in micro- and nanoscale devices by providing a detailed understanding of their molecular structure and the factors that control their structural, photophysical, and chemical properties.

## Accomplishments

Building on our earlier discovery of porphyrin nanotubes, which are formed by ionic self-assembly of two oppositely charged porphyrins in aqueous solution, we found many new members of this new class of nanostructures. By altering the molecular building blocks (porphyrins), novel nano- and microscale structural morphologies were found, and new ionic self-assembly methods were developed. The new porphryin nanostructures include nanofibers and fiber bundles, nanorod bundles, clover like microstructures, “strings of firecrackers,” microtubes, and nanospheres. All of these porphyrin structures have interesting absorption, fluorescence, resonance light scattering, electrical, and photocatalytic properties that make them potentially suitable as sensing elements of sensor arrays. For example, the porphyrin nanotubes are well formed, robust, hollow structures that exhibit interesting electronic and optical properties. Besides having structural characteristics making them suitable for incorporation into microdevices, we demonstrated that they are indeed sensitive to chemical vapors (e.g., pyridine, $NO_2$, acetic acid). Their chemical sensitivity potentially provides the functionality necessary for micro- and nanoscale sensing elements that rely on optical absorption, fluorescence and resonance light scattering, and electrical conductivity for gas phase detection.

Further research is necessary to fully exploit the wide range of new nanostructures that can be synthesized by the methods developed in this work and to advance the aim of using these nanostructures in microelectronic devices. The new porphyrin nanostructures and their physical properties revealed in this work demonstrate their utility in providing a new suite of components for the fabrication of nanosystems, such as ultrasensitive sensors for chemical and biological agents for counterterrorism and warfare, and for nanoscale electronic and photonic components, such as reconfigurable conductors, photonic lattice elements, alterable nanofluidic channels, and nanomechanical elements. As catalytic nanostructures, they also impact

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# Achieving a New Paradigm in Software Technology

*79883*

*G. C. Osbourn, J. W. Bartholomew, A. M. Bouchard*


## Abstract

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 always riddled with flaws ("bugs") that can cause unexpected failures. Our economy and national security have become increasingly dependent on such costly and unreliable software, and US industry relies increasingly on inexpensive software development in sensitive countries. This project will launch our ambitious effort to ultimately put an end to these serious and long standing problems. We seek to revolutionize how software systems are created for both US industry and government agencies using a new approach, called self-assembling software, which we demonstrated on a small scale. We propose 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 Department of Energy programs and dramatically alter the course of a trillion dollar world-wide industry. Our success will create a "disruptive" technology with enormous potential impact and economic value.


## Accomplishments

Our goal is to revolutionize the way that software is developed.

- *Milestone 1:* We designed 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). We are seeking protection of the intellectual property associated with the self-assembling building blocks and the overall environment design. We also designed and implemented our own compiler. This general purpose compiler that we built from tens of thousands of lines of Pentium 4 assembly code will directly and rapidly generate the building blocks that, in turn, self-assemble into large programs.
- *Milestone 2:* We reviewed the computer usability literature and attended a usability conference. The best available ideas for a next generation graphical user interface (GUI), along with innovations of our own, were incorporated into our completed GUI design.
- *Milestone 3:* We began implementation of the designs. Our Pentium 4 compiler, described above, is essentially fully functional. We wrote approximately 10,000 lines of code to implement the self-assembling building blocks and associated infrastructure. We also carried out our own usability tests on a mock-up of our GUI design ahead of schedule.
- *Milestone 4:* We are working on a new class of smart mutation mechanisms for computational evolution of self-assembly mechanisms.
- *Milestone 5:* We began to acquire feedback from other designers and consultants, a milestone originally planned for FY 2006.

# Nanopatterning of Electro-Active Polymers for Tunable Optics

*79884*

*D. W. Carr, G. R. Bogart*

## Abstract

Tunable optical devices for sensor applications have long been of interest to Sandia and its customers. These tunable systems enable new technologies for spectrometry, adaptive focal plane array imaging, beam shaping, and optical communications. There is a very promising technology that could make a substantial impact in this field, but it has yet to be explored for this application either within Sandia or externally. The use of electro-active-polymers (EAPs), also known as artificial muscle, could be ideal for tunable optical systems. Exploring this would require the development of a new field of micro- and nanopatterning of these polymer materials. Advances in this area could also lead to substantially new low-voltage actuation technologies that can enable novel microsystems.

In collaboration with John Rogers (Professor, University of Illinois Urbana-Champaign), we developed a process to pattern polyvinylidene fluoride (PVDF) films. A process for spin coating PVDF on glass and gold coated substrates was developed. We then created grating structures in this material using a nano-stamping process. The structures were poled, and we observed the piezo actuation with atomic force microscopy (AFM) and dynamic optical measurements. Further work is needed to adequately characterize the resulting materials and structures for applications in tunable optics, sensors, and actuators.

## Accomplishments

The technical approach focused initially on the integration of PVDF piezoelectric polymer with conventional microelectromechanical systems (MEMS). The PVDF film is modified through soft contact lithography (micromolding) to generate vias of approximately 0.25 µm in size. The film is a mixture of 80% PVDF and 20% polymethylmethacrylate that is used for better adhesion. Micrographs indicate a faithful transmission of the structure is made into the PVDF polymer. The grating structure is designed to be tested by high frequency photoacoustic resonance. This structure will be used to further characterize the dynamic physical properties of the material.

We developed a MEMS test structure to evaluate the functionality of the molded electroactive polymer structures. We developed a poling procedure and monitored the film deformation by AFM. The film returns to its original shape after the deformation voltage is turned off. This indicates that the MEMS testing structure and evaluation process was demonstrated for the production of these films. We also tried an all optical setup. We optically observed deformation, but determining the amount of deformation was limited by the signal-to-noise ratio of the system.

# Ultra-Cold Molecule Production

*79885*

*D. W. Chandler, L. A. Rahn*

## Abstract

The production of ultra-cold molecules is a goal of many laboratories throughout the world. We are pursuing a technique that utilizes the kinematics of atomic and molecular collisions to achieve the goal of producing substantial numbers of < 1 K molecules in a trap. A trap is an apparatus in a known location in the laboratory that holds a sample of molecules at a temperature below 1 K long enough to measure, and possibly further cool, the sample. We utilize collisions of an atom with a molecule that has a similar mass in order to slow the molecule. This technique is unique to Sandia. We built a trap and are in the process of utilizing it for the capture of slow molecules. We also bought a $CO_2$ laser for the purpose of trapping molecules.

## Accomplishments

We previously used a crossed molecular beam experimental apparatus to measure the differential cross section of the inelastically scattered NO molecules in NO + Ar collisions. A pair of doubly-skimmed pulsed free-jet expansions forms well collimated molecular beams that intersect at approximately 90 degrees. One of the beams is pure Ar, and the other is a mixture of 5% NO in Ar with NO molecules predominantly in the ground rovibrational state. The velocity mapped ion imaging technique is used to characterize the scattering distribution and density of scattered NO molecules. In this method, the elastically and inelastically scattered NO products are observed by quantum state selective ionization using (1+1') resonance-enhanced multiphoton ionization (REMPI) through the NO $A^2\Sigma \leftarrow X^2\pi$ transition at 226.057 nm. The bandwidth of our excitation laser is sufficiently narrow to ensure that only those NO molecules in a selected J state are excited to the A state and subsequently ionized by a 266 nm photon. The ions are formed in an electrostatic lens system that focuses and directs them onto a microchannel plate detector with a phosphorescent screen. Images created on the detector are captured with a charge-coupled device camera. Spatial positions in this image reveal that the velocities of the scattered NO molecules are being detected. The velocity is given by the ratio of the displacement on the detector, measured from the beam crossing point to the flight time of the ions from the laser/molecular beam intersection region to the detector, and multiplied by an instrumental magnification factor.

We extended these experiments to the $NH_3$ + Ne system. We obtained an image by ionizing the J=3 level of $NH_3$ utilizing the B (v=7)-X (1) transition at ~225 nm. The image shows a similar shape to the image of NO with Ar, with the distinctive build up of intensity around zero velocity in the laboratory frame of reference. Calculations indicate that the velocity distribution should be colder for the $NH_3$ than for the NO, but we are at the limit of our velocity measurement capabilities using the ion imaging technique so we do not observe colder $NH_3$. We built a trap, and we are attempting to trap these cold $NH_3$ molecules.

# Subsurface Measurements of Detonation Wave Structure

*79886*

*E. J. Welle, A. M. Renlund, K. J. Fleming*

## Abstract

Initiation and sustained detonation of explosives are strongly influenced by the material microstructure. Recently, modelers have evaluated the effects of shock wave phenomena within granular heterogeneous materials in the mesoscale regime. The most powerful experimental techniques used to quantify mesoscale phenomena are Doppler interferometry surface measurement techniques. These techniques provide critical information about the shock front, but only at the interface of the explosive using a mirror coated window. However, these interface measurements also have data corrupting boundary condition effects, which can lead to spatial smearing of the measurement resulting in coarser information about the explosive. The goal of this research effort was to evaluate the feasibility of conducting subsurface measurements on pressed granular beads and large single crystal explosives in an attempt to eliminate interface effects imposed by the current measurement techniques and therefore provide higher quality data.

To determine the feasibility of extending interferometer techniques to subsurface measurements, we had to acquire knowledge of optical properties of explosive crystals, modification of a velocity interferometry system for any reflector (VISAR), and rovibrational evaluation of other interferometer techniques and systems. To enable a broader execution of experimental activities, we established collaborations with researchers at Los Alamos National Laboratory (LANL) and BWXT Pantex. These collaborations led to the supply of large single crystals of HMX and HNS, commonly used explosives. Additionally, collaboration with LANL researcher David Holtkamp led to the evaluation of a photon Doppler velocimetry (PDV) system that was expected to be more ideally suited for subsurface measurements than a traditional VISAR system.

## Accomplishments

Our efforts progressed from an idea, to solidifying that idea, to building necessary test hardware, to collecting supporting data, and to conducting experiments to evaluate feasibility. The basic concept of this effort centered on collecting subsurface interferometry data in a granular bed or in a large single crystal of explosive. This information is important in granular beds because it would provide invaluable data about detonation wave structure. For single crystals, these data would allow definition of critical shock characteristics that can be used to increase the accuracy of models and to enhance fundamental understanding.

We began with a trip to Lawrence Livermore National Laboratory (LLNL) to discuss optical diagnostic techniques currently being evaluated. More specifically, we discussed a technique called PDV. Fundamentally, the PDV technique relies upon resolving “beat frequencies” imposed upon unshifted light by the Doppler shifted light returning from a target. The PDV technique appeared to be an ideal candidate for conducting subsurface measurements in explosive media because it uses a 1550 nm pump beam, its dynamic range of measurable velocities is only limited by the speed of the detectors and oscilloscopes, and it requires very low levels of return light to resolve velocity information.

Next, we focused on collecting relevant explosive data and explosive materials and attempting to collect the necessary hardware to construct a near infrared (NIR) VISAR system. A NIR system was ideal because of the decreased scattering, or increased penetration, characteristics of longer wavelengths of light relative to the standard VISAR wavelengths that are in the visible portion of the spectrum. This path was ultimately abandoned for two primary reasons: (1) resurrection of an appropriate long pulsed 1064 nm laser appeared to be unlikely, and (2) after a somewhat exhaustive review of NIR photo detectors, a suitable low cost and high speed 1064 nm detector could not be identified. This resulted in the decision to use a standard VISAR wavelength and investigate other avenues for evaluating NIR interferometry systems.

Dave Holtkamp visited our facility to conduct measurements on explosive plane wave generators using a PDV system and collected some curious results during that testing. After discussing his results, we believed that subsurface measurements had been unknowingly achieved.

We set up follow-on experiments including parallel evaluations using VISAR and PDV on pressed pellets of HNS and TNT as well as single crystals of HNS and HMX. We pressed HNS and TNT pellets at low and high density conditions to evaluate the feasibility of collecting measurements across a range of densities. The highest density conditions for the two explosives were 97 and 91 percent of the theoretical maximum crystal density (TMD) for TNT and HNS, respectively. Using PDV, we successfully replicated the initial data. The data suggested that subsurface detonation wave phenomena were being probed; however, this was only successful in the high density TNT samples and not in the HNS or low density TNT samples. Based upon these results, a limitation of this type of measurement in a granular bed may be that the samples are constrained to high densities near TMD. Unfortunately, we did not conduct measurements in single crystals. Future testing should include further evaluation of the VISAR and PDV techniques using pressed pellets and single crystals. In summary, the experimental results suggest that subsurface measurements may be possible in granular beds. However, further confirmation is necessary, along with a mapping of the possible measurement space.

# Assembling Semiconductor Nanocomposites Using DNA Replication Technologies

*79887*

*G. D. Bachand, K. K. Crown, B. W. Heimer*

## Abstract

Recent pioneering work at Sandia demonstrated the ability to synthesize assemblies of functionalized, CdSe nanocrystal quantum dots (nQDs) using biologically based replication strategies. To date, small clusters of nQDs (less than 500 nm in size) have been assembled using deoxyribonucleic acid (DNA) polymerase to incorporate CdSe particles, in a sequence specific manner, into newly synthesized DNA strands. Our long term goal is to use DNA replication technologies to assemble two- and three-dimensional nanocomposite structures composed of DNA scaffolds and semiconductor or metallic nanoparticles precisely arrayed in a programmed manner upon those scaffolds. In FY 2005, the primary objectives of this project were to demonstrate the attachment of nQDs to DNA nucleotides using varying linkages, to characterize the incorporation of modified DNA nucleotides in DNA replication, and to demonstrate the assembly of 15 micron nQD-DNA assemblies.

We evaluated a variety of covalent attachment chemistries to link nQDs to DNA strands. Experimental data suggested that incorporation of nQD-modified DNA nucleotides during the replication was not possible. The results were consistent with published work on crystal structures as well as the biophysical properties of the enzyme. Incorporation of amine-, thiol-, and biotin-modified DNA nucleotides was demonstrated and characterized with respect to the efficiency of incorporation. Finally, using fluorescence microscopy, we demonstrated the ability to synthesize 15 micron DNA strands with attached nQDs. Overall, we achieved a number of significant technical advances during the course of this project and opened the door to continued development of large scale manufacturing processes of photonic materials using DNA replication technology.

## Accomplishments

FY 2005 work aimed to demonstrate the assembly of patterned arrays of nQDs using 15 micron long DNA templates and DNA replication technologies. Three specific technical objectives were addressed: (1) demonstrate the attachment of nQDs to DNA nucleotides using varying linkages, (2) characterize the incorporation of modified DNA nucleotides in DNA replication, and (3) demonstrate the assembly of 15 micron nQD-DNA assemblies. Overall, excellent progress was made with respect to these objectives.

In terms of the first technical objective, we evaluated three attachment chemistries with respect to linking DNA nucleotides to nQDs and/or fluorescent dyes. Initially, work focused on noncovalently linking DNA nucleotides (biotin-14-2'-deoxycytidine-5'-triphosphate [biotin-dCTP]) to nQDs via biotin-streptavidin interaction. Although nQD attachment was successful, the noncovalent linkage was not able to withstand the temperature necessary for DNA replication. Next, work focused on coupling fluorescent dyes to DNA via covalent linkages. Three modified DNA nucleotides were evaluated: 2-amino-2'-deoxyadenosine-5'-triphosphate [2-amino-dATP], 2'-deoxycytidine-5'-O-(1-thiotriphosphate) [thio-dCTP], and 5-aminoallyl-2'-deoxycytidine-5'-triphosphate [aminoallyl-dCTP]. The results suggest successful attachment to thio-dCTP nucleotides via maleimide chemistry. Attachment to amine modified nucleotides was less successful. In addition, experimental data suggested that incorporation of nQD-modified or dye-modified DNA nucleotides during the replication was not possible. The results are consistent with published crystal structures as well as the biophysical properties of the enzyme.

The second objective focused on characterizing the incorporation of modified DNA nucleotides into newly synthesized DNA strands. A circular DNA plasmid that contained the coding sequence for a monomeric kinesin was utilized as the DNA template in these experiments. The polymerase chain reaction (PCR) method of DNA amplification was to synthesize a 1.3 kilobase DNA strand with the modified nucleotides. We quantified the relative efficiency of amplification by measuring the mass of the resulting DNA bands using agarose gel electrophoresis. Overall, the efficiency of incorporation was significantly dependent on the relative ratio of modified to unmodified nucleotide in the reaction mixture. This result suggests the DNA polymerase is

able to relax its intrinsic specificity for DNA nucleotides and permit efficient incorporation of modified nucleotides. Optimal incorporation of all modified nucleotides was observed at a ratio of 3:1 modified to unmodified nucleotides. The thio-dCTP was incorporated with the greatest relative efficiency (13%), followed by 2-amino-dATP (9%), and biotin-dCTP (5%). The aminoallyl-dCTP had a slight inhibitory effect on the PCR process, as seen by a relative efficiency of incorporation of -8%.

The final technical objective was to demonstrate the assembly of 15 micron nQD-DNA assemblies. We used a modified version of the PCR methodology to accomplish this task. Specifically, we used twelve antisense DNA oligomers to “prime” the synthesis of a single stranded lambda DNA molecule and incorporated biotin-dCTP nucleotides in the process of synthesis. Streptavidin coated quantum dots were then attached to the DNA strands (pre-stained with a fluorescent dye); nQD-DNA assemblies were then observed by epifluorescence microscopy. The results demonstrated successful attachment of nQDs to 15 micron long lambda DNA strands. Further, the attachment of nQDs to DNA strands was relatively low (< 20 nQDs per strand), which is consistent with the relatively low rate of biotin-dCTP incorporation previously observed.

In summary, the critical technical objectives proposed for FY 2005 were successfully achieved. Key methodologies and proof-of-principle experiments established a core technology for assembling semiconductor quantum dots on DNA using various chemical linkages.

# Building Cognition on Biology: Self-Organization in the Brain

*79888*

*A. Backer, A. Slepoy, R. J. Pryor*


## Abstract

Saliency detection in images is an important outstanding problem both in machine vision design and the understanding of human vision mechanisms. Recently, seminal work by Itti and Koch [1] resulted in an effective saliency detection algorithm. The algorithm implements current methods in image compression to perform a bootstrap search for saliency points. We want to reproduce the original algorithm in a Vision software application and explore its limitations. We propose extensions to the algorithm that promise to improve performance in the case of "difficult to detect" objects.


## Accomplishments

A variety of real and computer generated images were tested on the algorithm. The results indicate that the algorithm detects locations of interest in the image in a manner that makes functional sense. However, it should be noted that it is difficult to objectively define criteria for its performance on such images. Although information exists on visual scanpaths (the paths a subject's eyes follow when confronted with an image), it is unclear to what extent the precise trajectory followed is related to covert (bottom-up) shifts of attention or overt (top-down) shifts [1].

Itti and Koch [1] devised a test involving 44 images of camouflaged military vehicles in a natural setting. They compared the algorithm to 62 human observers and measured the number of attentional shifts the algorithm took before finding the vehicle. On average, the algorithm found the target in less time than the human observers.

General structure of the algorithm is instructive. It is constructed of several sequential components:

(1) Channel splitting and analysis.
(2) Scale splitting and analysis.
(3) Competitive selection.
(4) Synthesis of scales.
(5) Synthesis of channels.
(6) Competitive selection.

We identified aspects of automatic model construction and subsequent application in a bootstrap fashion. Model construction consists of partitioning/segmentation of information and extraction of the dominant features. The model is then constructed by recombination of the dominant features and exercised via a competitive selection mechanism. Competitive selection is reminiscent of a global optimization procedure.

We explored adding more channels to increase the capabilities of the algorithm. For example, a stereo channel would be instrumental in breaking camouflage. A motion channel, for movies instead of static images, could also be included to enable object tracking. It is well established that the human eye is incredibly sensitive to motion. Other static channels such as infrared can be added as well.

We found that much of the technology applied here has strong parallels in the field of computational modeling of materials and systems, particularly in the field of multi-scale modeling. We hope to use these parallels to develop original approaches and insights into these important areas.

[1] L. Itti, C. Koch, "A Saliency-Based Search Mechanism for Overt and Covert Shifts of Visual Attention," *Vision Research*, vol. 40, no. 10–12, pp. 1489–1506, May 2000.

# Nanodielectrokinetic Chromatography (nanoDEKC): Rapid Separation and Classification for Biodefense and Biomedical Applications

*79889*

*G. S. Chirica, G. J. Fiechtner*

## Abstract

We demonstrated a new approach to rapid and selective concentration of particles from liquid samples using a miniaturized point detection device. We explored and optimized the fabrication of a simple, cost-effective, and extremely versatile system that extends the testing capabilities of chip based devices currently used for continuous water monitoring applications. A range of samples with various sizes and compositions were tested to assess the optimal operating range for selective manipulation of microparticles. In a simple experimental setup, direct connection to a detection device delivered minimal dilution of the concentrated plug. Imaging data illustrate non-linear electrohydrodynamic effects induced in our system generated attractive and repulsive forces that amounted to controlled trapping, streaming, and/or release of particles.

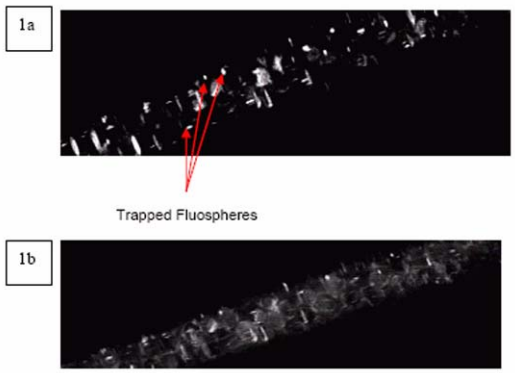
The adsorptive effects of samples containing various microorganisms (viruses, spores, vegetative cells) were tested on materials used for chromatographic applications. In particular, we identified the main parameters that govern fractionation of microorganisms, proteins, and low molecular weight species as well as the concentration of viral particles and spores using chromatographic media. We observed the compatibilities and limitations of coupling dielectrophoresis and chromatography in dielectrokinetic chromatography (DEKC), a two-dimensional separation technique.

This study presented an additional advantage of our system: its suitability for testing dielectrophoretic effects on nanoscale dimensions at a cost that is substantially lower than that for chip based devices. We evaluated novel matrices for testing non-linear effects exerted around nano size features. Preliminary results and the breakthrough potential of nano-dielectrophoresis (nano-DEP), particularly in combination with chromatography, are presented.

## Accomplishments

We demonstrated the feasibility of a new system for rapid concentration and separation of vegetative cells, spores, viruses, and other particulates. We used surprisingly simple instrumentation: fused silica capillaries packed with a variety of materials to generate steep electric field gradients able to control the movement of micro and nanometer size particles. The versatility of our system allows exploration of new areas, such as nano-DEP and DEKC, which are unavailable to other devices.

Our experimental setup enabled direct connection to a detection device with minimal dilution of the concentrated plug. This allowed imaging of the behavior of particles with diameters ranging from 100 to 1000 nm present in the liquid sample. We examined the effect that packed bead size, electric field strength, and liquid composition have on streaming, trapping, or release of particles. For example, we imaged the trapping of 1 µm fluorescently labeled particles in a 100 µm inner diameter capillary packed with 45 µm polystyrene beads as electric fields of 45 V/mm were applied. After one minute, the voltage potential is dropped and the trapped beads are released in a concentrated plug. We also demonstrated rapid trapping and release of microorganisms such as the fluorescently labeled *Bacillus subtilis* spores.




*Imaging of trapping and releasing of fluorescent particles in the non-uniform field gradients generated in a packed capillary.*

In order to identify the materials with potential for use as media for nano-DEP, we surveyed several packings with respect to their ability to generate steep elec-

tric fields gradients and their suitability for use in our experimental system. We studied porous materials in detail and assessed the effect of liquid composition, bead size, and, particularly, material composition on separations. The data show conditions in which adsorptive interactions are minimized to allow high recovery size based separation of microorganisms from molecules. We demonstrated microscale separation of bacteriophage T4 from a small peptide in less than two minutes. This study revealed the best materials in which nano-DEP forces could be observed and studied.

Coupling methods with specific optimal operating parameters and limitations to achieve synergic separation is not a trivial task. One has to thoroughly investigate the methods (in our case with respect to capabilities for microorganism analysis) and observe the compatibility domains that enhance the individual performance of each technique. The literature that investigates chromatography of microorganisms is very scarce. Therefore, we focused a part of our study on understanding the basis of adsorptive interactions exerted on bioparticles by various chromatographic packings. This resulted in a demonstration of the capture and release of viruses from an ion-exchange material. More significantly, we demonstrated that the method can be used quantitatively with high sample capacity.

In conclusion, this project demonstrated the feasibility of concentrating nano and micro sized particles in a simple, cost effective, and versatile system. We studied the key parameters responsible for microparticle manipulation in our device. Non-conventional materials were analyzed to determine their suitability for nano-DEP. We identified compatibility domains between dielectrophoresis and chromatography by testing the capability of each method with respect to bioparticle interaction. This work proved our hypothesis and generated the necessary information for extending research in directions that can be easily explored using our system.

## Refereed

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# Quantum Computing Using Interacting Semiconductor Quantum Wires

*79890*

*E. S. Bielejec, M. P. Lilly, J. L. Reno*

## Abstract

The quantum transport group at Sandia demonstrates world-leading, unique double quantum wire devices where we have unprecedented control over the coupling strength, number of one-dimensional (1-D) channels, overlap, and interaction strength in the nanoelectronic system. In this project, we propose to study 1-D/1-D tunneling with the ultimate aim of preparing and detecting quantum states of the coupled wires. In a region of strong tunneling, electrons can coherently oscillate from one wire to the other. By controlling the velocity of the electrons, length of the coupling region, and tunneling strength, we will attempt to observe tunneling oscillations. This first step is critical for further developing double quantum wires into the basic building block for a quantum computer and for other coupled nanoelectronic devices that will rely on coherent transport.

## Accomplishments

We studied resonant tunneling measurements between vertically coupled double quantum wires fabricated on both sides of a double quantum well GaAs/AlGaAs heterostructure. We devised a tunneling geometry that allows for well defined confinement potentials for the 1-D wires, as well as allowing for the independent formation and control of the number of occupied subbands in the individual wires. Resonant tunneling occurs when an electron in one wire tunnels to the other wire, conserving both energy and momentum. We compared 2-D/2-D tunneling to the tunneling results in the 1-D/1-D regime. We studied this tunneling system in double quantum wire devices with different coupling strengths (varying the tunneling barrier between the independently contacted layers) and different wire lengths. By minimizing the 2-D/2-D component of the tunneling, we were able to identify and characterize the 1-D/1-D tunneling features as a function of density and parallel field.

The 1-D density is directly controlled in each wire using the split gates. Independent contact to each wire is achieved using the depletion gates. Signatures of multiple 1-D subbands are clearly observed at high magnetic fields. While some qualitative comparison to tunneling theory for non-interacting electrons is successful, we note a number of deviations as well. Most significantly, the structure at low fields for a single 1-D subband is more complicated than that observed for 2-D tunneling. We also observe broad resonant features at all 1-D densities when a much narrower and well defined resonance is expected. To further our understanding of the deviations, we are considering finite size effects and the role of many-body Coulomb interactions in the double quantum wire system.

In summary, 1-D tunneling in a split gate defined double quantum wire was measured for the first time. The split gate provides explicit control over the density and number of occupied subbands in each wire. The experimental tunneling spectroscopy for the 2-D system is in good agreement with theory and provides a demonstration of the technique. A number of features in the 1-D spectroscopy can be clearly explained using non-interacting pictures for the electrons, but other features cannot be so easily understood. Interactions within and between the wires is one possible explanation. Due to the significant difficulties in preparing a single wire system for tunneling studies, using double quantum wires as a platform for quantum computing will be extremely difficult. Other solid-state approaches such as coupled quantum dots will be more feasible to develop into a qubit.

## Refereed

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# Cell Directed Assembly of an Integrated Nanoelectronic/Nanophotonic Device for Probing Cellular Responses on the Nanoscale

*79891*

*C. J. Brinker, S. Singh, C. E. Ashley, H. K. Baca, E. C. Carnes*

## Abstract

Our discovery that the introduction of living cells (*Saccharomyces cerevisiae*) dramatically alters the evaporation driven self-assembly of lipid-silica nanostructures, suggested the formation of novel bio/nano interfaces useful for cellular interrogation at the nanoscale. This one year "out of the box" project focused on the localization of metallic and semi-conducting nanocrystals (NCs) at the fluid, lipid-rich interface between *S. cerevisiae* and the surrounding phospholipid-templated silica nanostructure, with the primary goal of creating surface enhanced Raman spectroscopy (SERS)-active nanostructures and platforms for cellular integration into electrode arrays. Such structures are of interest for probing cellular responses to the onset of disease, the understanding of cell to cell communication, and the development of cell based biosensors. As SERS is known to be sensitive to the size and shape of metallic (principally gold and silver) NCs, various sizes and shapes of NCs were synthesized, functionalized, and localized at the cellular surface by our "cell directed assembly" approach. We performed laser scanning confocal microscopy and in situ grazing incidence small angle x-ray scattering (GISAXS) experiments to study metallic NC localization. We conducted preliminary Raman spectroscopy studies to test for SERS activity. We used interferometric lithography to construct high aspect ratio cylindrical holes on patterned gold substrates and performed electro-deposition experiments in a preliminary attempt to create electrode arrays. We developed a new printing procedure for cellular integration into nanostructured platforms that avoids solvent exposure and may mitigate osmotic stress.

## Accomplishments

Cryo-transmission electron microscopy (TEM) and confocal fluorescence microscopy studies employing optically labeled 2 nm gold NCs showed that, during evaporation induced self-assembly of lipid/silica nanostructures incorporating *S. cerevisiae*, gold NCs are localized at the cellular surface and eventually imported into the cells. As NC localization could allow the formation of a SERS-active nanostructure that might be operative even during internalization, we focused on extending our initial work to creating SERS-active structures in which living cells and NCs are immobilized in a nanostructured fluidic architecture. SERS is known to be sensitive to the size and shape of the metallic NCs, with 20–50 nm gold or silver NCs being optimum. As our initial work employed 2–3 nm gold NCs, we followed literature procedures to synthesize larger gold NCs (5 nm spheres), silver NCs (30 nm spheres), and low aspect ratio, gold nanorods (80 x 40 nm).

Surface functionalization is important in directing NC localization. For our initial studies using 2–3 nm gold NCs, we modified the initially hydrophobic alkanethiol terminated surfaces with phospholipids (and optically labeled phospholipids), creating a hybrid bilayer that made the NCs water soluble and directed their localization. Similar procedures were used for larger gold NCs and hydrophobic oleylamine terminated silver NCs. Gold nanorods, however, are prepared in aqueous solvents with cetyltrimethylammonium bilayer terminated surfaces. We did not develop a satisfactory means to functionalize these NCs with phospholipids.

To study NC localization, we used scanning electron microscopy (SEM), TEM, GISAXS, and, for optically labeled NCs, fluorescence microscopy and confocal laser scanning microscopy. These studies, although limited in the scope of the functionalization chemistry, suggested that phospholipid functionalization is necessary for cellular localization. Interestingly, the GISAXS studies indicated that NC localization can, in some cases, result in ordered NC arrays at the cellular surface. Such arrays, if SERS active, could be precisely tuned with respect to particle spacing, perhaps allowing optimization of the SERS-active nanostructure. SEM studies performed with energy dispersive electron spectroscopy confirmed that fluorescence microscopy results show NC localization at the immediate surface of the cell.

Having confirmed cell directed NC localization at the cell surface, we made attempts to obtain SERS

enhanced spectra. As SERS is notoriously sensitive to both sample preparation (e.g., size and concentration of NCs) and the operating conditions (e.g., excitation wavelength), our group's lack of access to a dedicated Raman spectrometer seriously impeded our progress in this area. Our collaborations with the University of California, Santa Cruz have so far resulted in non-reproducible results. We presume this is because, with a limited number of experiments, the heterogeneous nature of our samples (cell-containing regions separated by silica nanostructures), combined with the sensitivity of SERS, makes it difficult to achieve reproducibility. Experiments performed at Sandia allowed us to obtain Raman spectra of samples containing cells with and without localized NCs. However, further tests are needed to confirm the SERS enhancement of these spectra, as well as to optimize the parameters for performing SERS.

A concurrent task of this project was the construction of a substrate containing small, high aspect ratio gold posts for use as electrodes. The idea was that cells would recognize and internalize lipid coated gold posts, thereby integrating themselves into an electronic platform. Interferometric lithography using SU-8 epoxy was chosen as a lithography system starting point. We optimized exposure/develop parameters that yield hole aspect ratios of > 10. Although SEM examination, after electrodeposition of the films, indicated successful electrodeposition, attempts at physical removal of the crosslinked resist caused the deposited posts to break away from the substrate. In order to address these issues, a different negative photoresist system that can be chemically dissolved is currently under investigation.

## Refereed

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## Other Communications

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C.J. Brinker, "Bio-Inspired Self-Assembly of Porous and Composite Nanostructures," presented at the AFOSR Biomimetics, Biomaterials and Biointerfacials Sciences Program Review, San Diego, CA, January 2005.

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H. Baca, C. Ashley, E. Carnes, D. Lopez, S. Singh, and C.J. Brinker, "Cell-Directed Assembly of the 3-D Bio-Nano Interface," presented at the MRS Multifunctional Ceramic Composites Workshop, Urbana, IL, October 2005.

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H. Baca, C. Ashley, E. Carnes, D. Lopez, S. Singh, and C.J. Brinker, "Complex Structures and Functions Through Cell-Directed Assembly," presented at the 13th International Workshop on Sol-Gel Science and Technology, Los Angeles, CA, August 2005.

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# Self-Assembling Holographic Biosensors and Biocomputers

*79892*

*J. S. Schoeniger, Y. K. Light, A. M. Trent, G. D. Bachand*

## Abstract

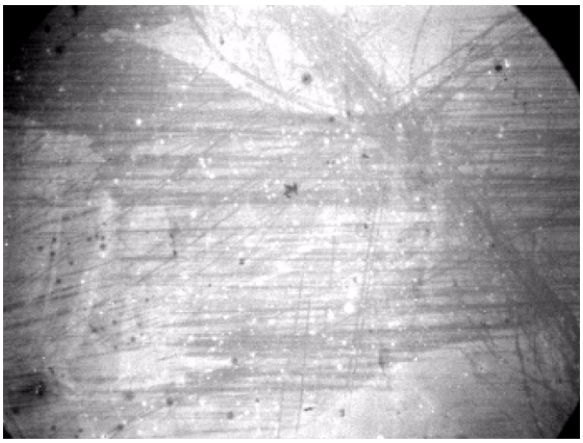
Molecular computers are becoming a reality. However, advances still need to be made in interfacing molecular computers to the macroscopic world. Sensors that turn molecular binding events into macroscopic signals through self-assembly phenomena have been demonstrated. These sensors essentially perform signal transduction and amplification. The work proposed will explore the feasibility of integrating nanopatterning, molecular motors, and biophotonics to produce devices that can relay information about molecular events as diffraction patterns. We propose a new solution to the nano-macro interface problem, as well as the integration of multiple modalities, employing microtubules (MTs) (self-assembling rod-like motile proteins).

We propose that nanopatterned kinesin can be used to cause MTs to assemble to form diffraction gratings in response to the presence of adenosine triphosphate (ATP). This creates a basis for making diffractive optical elements that can rearrange in response to optical, electronic, or chemical signals. Necessary to testing this device concept is the ability to pattern kinesin features with submicron resolution in a manner compatible with preserving kinesin activity. Also, other regions of the surface must be rendered biocompatible but non-interactive with kinesin such that the preferred location for MTs is on top of the kinesin features. Various techniques for protein patterning were considered, and we developed a new approach based on stamping. We present results showing successful patterning of kinesin and surface passivation using conditions compatible with preserving kinesin activity.

## Accomplishments

A concept underlying this project is that if kinesin, the motor protein that interacts with MTs, can be patterned in lines with submicron width and separation and the spaces between the lines of kinesin are made to have lower binding energy with tubulin than kinesin, the MTs would migrate onto and align with the kinesin "tracks." Previous work by one of the investigators demonstrated that MTs can be "decorated" with substances that could increase their effective index of refraction. Thus, migration of MTs onto such a kinesin array would cause the assembly of a diffraction grating in response to the presence of ATP. This enables the implementation of optical signal transduction and feedback schemes that could be used for chemical sensing and optical computing.

The project's first objective was to find a means of nanopatterning kinesin that would preserve its enzymatic activity (ATPase) and its motor protein properties and also allow interdigitated patterning of a biocompatible, non-adherent layer to pattern an optical grating. This turned out to consume the efforts of this exploratory project, although we believe we were ultimately successful.



*Fluorescence micrograph (200x) of kinesin self-assembly onto nanopatterned BMPS, 400 nm lines nominal.*

A general strategy for the patterning of active enzymes requires that they be maintained in aqueous buffers. Although proteins can be spotted using robotic pin-based microarraying technology or covalently attached to photo-patterned arrays or photo-activatable linkers, these techniques cannot generally produce submicron features. On the other hand, proteins can be directly stamped using poly-di-methyl siloxane (PDMS) polymer stamps with submicron resolution, provided that they are "dry" stamped. Because PDMS stamping has been shown to be useful for nanopatterning in a number of contexts, we adopted this technology as our means of patterning.

We first demonstrated that kinesin could be covalently attached to silica surfaces. Recombinant *Thermomyces lanuginosus* kinesin with a single unpaired cysteine was expressed and purified and shown to be active upon ATPase assay. Porous silica beads with amino surface groups were prepared. These were reacted with beta-maleimidopropionic N-hydroxysuccinimide ester (BMPS) to create a cys-reactive maleimide surface and incubated with fluorescently labeled kinesin. The washed beads were then subjected to total hydrolysis in concentrated base. Protein attachment was demonstrated by the presence of excess fluorescence versus controls without BMPS.

We next demonstrated that a PDMS stamp inked with fluorescently labeled kinesin could be used to pattern large scale (100 micron) lines on amino-silane/BMPS treated glass slides. Although this demonstrated that kinesin could be selectively wet stamped, efforts at nanopatterning using a PMDS grating stamp with a 400 micron pitch failed, presumably because diffusion of the protein obliterated the pattern.

We were able to successfully nanopattern the kinesin as follows:

We inked the PDMS stamp with a BMPS solution, which was allowed to dry on the stamp. We then dry stamped the BMPS onto the slide, and solid state reaction of the BMPS with the amino groups took place. We washed the slide with low pH buffer to block *N*-hydroxysuccinimide (NHS) ester reactivity while we removed excess BMPS. We then incubated the slide with fluorescent kinesin overnight. After rinsing, the slide was reacted with NHS-PEG to passivate the areas not reacted with kinesin. Although scratches distorted the nanopatterned image, we clearly observed a series of fine lines. Analysis using confocal fluorescence microscopy and atomic force microscopy should elucidate the exact nature of the patterns produced.

While not definitive, results suggest that kinesin can be successfully nanopatterned by self-assembly onto PDMS stamped linkers. Further development of orthogonal linker chemistries should allow interdigitated lines of protein and polyethylene glycol to be patterned without intermixture or covalent modification of the protein. While the amount of protein patterned did not allow ATPase activities to be measured, optimization of the kinesin attachment protocol should enable fully active protein to be patterned as assayable by MT motility assays.

# Optical Diagnostics for Aerosols and Other Particles

*80597*
*H. A. Michelsen*

## Abstract

Lack of understanding of the optical properties of aerosols produces the greatest uncertainty in climate change models. Atmospheric soot particles could lead to either heating or cooling of the atmosphere, depending on their absorption and scattering properties. Although the optical properties of dry soot particles have been studied extensively, the uncertainties on the quantitative parameters are still very large. Studies on coated particles are limited, and the optical properties of these more complex particles are even more uncertain. This project aimed to measure the scattering and absorption of light by submicron particles and focused on combustion generated particles of atmospheric relevance. An apparatus was built to generate and coat soot particles under controlled conditions in the laboratory. This apparatus also included an optical detection region to measure extinction cross sections by cavity ringdown spectroscopy and angle- and polarization-resolved scattering.

Students performed this project as part of the Harvey Mudd Physics Clinic. Students successfully created soot particles in the lab by partially combusting ethylene. They collected and imaged some of these particles using scanning electron microscopy (SEM) to analyze their structure. They designed and constructed a coating system but did not test it. They designed and constructed the ringdown cavity and angle-resolved scattering systems. They detected no signal using the cavity ringdown equipment, possibly because of problems with the laser source and detector. Testing utilized both a diode and a helium-neon laser, and two different photodetectors, but the cavity ringdown experiment is still non-functional. They successfully tested the angle-resolved scattering experiment and made preliminary calibration measurements.

## Accomplishments

The goal of the current project was to build a system to measure optical properties of coated soot particles under controlled conditions. Physics students participating in the Harvey Mudd College Clinic Program designed the system to measure total particle extinction and multi-angle elastic scattering. The students selected cavity ringdown spectroscopy (CRDS) as the technique to measure extinction cross sections of the low density, weakly absorbing, and scattering sample. They designed a second experiment to measure scattering as a function of angle with a 5–10 degree angular resolution.

The students designed and built the main components of the system and performed preliminary experiments. They generated and sampled soot, then imaged the soot SEM. They built the cavity for the CRDS experiments but did not observe a CRDS signal before the end of the academic year. They built and tested the scattering chamber but did not calibrate it.

- Experimental Design

Soot aerosols are produced by the partial combustion of ethylene in a co-flow diffusion burner. A valve on the exhaust forces a small flow of the air-soot mixture down the flow path to the optical experiments. A diffusion dryer removes water vapor to prevent additional scattering and absorption during measurements. Particles are coated by sending the flow through a heated flask containing the coating of choice. The flow conditions are carefully controlled to maintain constant particle density in the detection regions during optical measurements.

The ringdown cavity consists of two aligned spherical mirrors with a radius of curvature considerably greater than their separation. We focused our attention on continuous wave (CW) CRDS, in which a CW laser beam is incident on one of the cavity mirrors. If the frequency of the light is such that an integral number of waves fit inside the cavity, a standing wave will form, and the intensity of the light in the cavity will increase as more light enters. In an empty cavity, the only loss of intensity will be from transmission through the two mirrors. Absorption of other components inside the cavity will lead to higher loss. As soon as the incident beam is stopped, the intensity loss will take the form of a single exponential decay with a decay rate that depends on the absorption within the cavity. By comparing the signal decay time with particles in the cavity to that for the empty cavity, a measurement of the total extinction cross section of the particles may be made.

The apparatus for the scattering measurements consists primarily of a rotatable arm attached to the flow tube. This arm includes imaging optics, a polarizer, and an optical fiber to couple the light to a photodiode used as a detector. The arm is placed in a box that is purged with a slow flow of air to prevent air from the flow tube to leak into the room, and the optical fiber is used to bring the signal to the detector outside the purge box.

In the scattering calibration, the helium-neon laser provided light to scatter from polystyrene latex spheres (PLSs) with 500 nm diameter. The PLSs acted as approximate Mie scatterers for calibration. The setup was first aligned without the PLS solution in place. The input light was linearly polarized using a polarizing filter. Scattering of the polystyrene spheres in water was recorded as a function of angle.

# Supramolecular Structures of Peptide-Wrapped Carbon Nanotubes

*84269*

*A. L. Frischknecht, S. L. Rempe, M. G. Martin*


## Abstract

Carbon nanotubes (CNTs) are unique nanoscale building blocks for a variety of materials and applications, from nanocomposites, sensors, and molecular electronics to drug and vaccine delivery. An important step towards realizing these applications is the ability to controllably self-assemble the nanotubes into larger structures. Recently, amphiphilic peptide helices have been shown to bind to CNTs and thus solubilize them in water. Furthermore, the peptides then facilitate the assembly of the peptide wrapped nanotubes into supramolecular well aligned fibers. We investigated the role that molecular modeling can play in elucidating the interactions between the peptides and the CNTs in aqueous solution. Using ab initio methods, we studied the interactions between water and CNTs. Classical simulations can be used on larger length scales. However, it is difficult to sample large biomolecules, such as the amphiphilic peptide of interest here, in atomistic detail. Thus, we explored both new sampling methods using configurational bias Monte Carlo simulations and coarse-grained models for peptides described in the literature. An improved capability to model these inorganic/biopolymer interfaces could be used to generate improved understanding of peptide-nanotube self-assembly, eventually leading to the engineering of new peptides for specific self-assembly goals.


## Accomplishments

The original goals of this “out of the box” project were to investigate the mechanism that binds the engineered peptide nano-1 to CNTs using molecular modeling, and then to build a phenomenological model of the peptide-nanotube self-assembly. These goals proved to be too ambitious for a one year research project. However, we made significant progress towards the modeling of peptide-CNT complexes in aqueous solution.

We began by investigating the force fields available in the literature to simulate peptide/CNT interactions and implemented these in the Monte Carlo code Towhee. Ab initio calculations focused on understanding the interaction between water and CNTs, due to controversy in the field about this interaction. We calculated the interaction of water with the exterior of the CNT and found a similar value to that of water inside a CNT. Due to the complexity and large size of the nano-1/CNT complexes, we conducted a brief survey of the literature on coarse grained models for peptides. There have been many different approaches to modeling peptides explored in the literature. Most of these models would require more time to implement than was available for the current project, but knowledge of them is useful for future work.

The major accomplishment for the year was the development and implementation of new algorithms to sample peptide conformations. We developed a new formulation of configurational bias Monte Carlo using arbitrary distributions to generate trial bond lengths, angles, and dihedrals. The formulation provided similar acceptance rates with substantially less computational effort. When combined with an alternative formulation of coupled-decoupled configurational bias Monte Carlo, significant improvements in the acceptance rate are obtained for cyclic molecules. This allows the simulation of complex biomolecules such as polypeptides and deoxyribonucleic acid that both contain a significant number of cyclic subunits. When tested on the peptide nano-1, we were able to grow the side chains using the new algorithms, given an initial configuration for the backbone atoms.

# Robust Spore Based Detection System

*84270*
*T. W. Lane, J. N. Kaiser*

## Abstract

Cell based biosensors have the potential for exquisite selectivity and sensitivity, yet the challenges associated with keeping cells viable in a detector system are profound. Some bacteria produce spores that are remarkably resistant to extremes of temperature and desiccation and are stable for years without refrigeration. The goal of the proposed research is to create a robust cell based detection system using signal transduction processes found in bacterial spores.

Bacterial spores typically germinate to form vegetative cells upon addition of specific chemical germinants. Upon binding of the germinant to receptors on the spore, the first step in germination is release of dipicolinic acid, followed by amino peptides and esterase enzymes. We will 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, it will elicit the release of many molecules of dipicolinic acid. The molecules can be directly assayed or assayed using esterases that will convert a specific non-fluorescent reagent to a fluorescent one. Either of these scenarios constitutes 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 step (proof-of-principal) is the creation of receptors with altered specificity.

Since spores remain dormant until activated by analyte/germinant, this system will be stable over a range of temperatures, resistant to desiccation, and not require a continuing supply of growth medium to maintain the viability of the biological detector system.

In FY 2005, we cloned the gerA operon from *Bacillus subtilis* and developed a spore germination assay suitable for high throughput screening of germination mutants. We obtained a strain of *Bacillus subtilis* containing a deletion of the gerA operon and tested this mutant with our germination assay. We are currently creating our first variant receptors by error prone polymerase chain reaction (PCR) and gene shuffling.

## Accomplishments

Initially, we planned to generate diversity in the germination receptors solely through the creation of chimeric receptors created by swapping sequences with receptor genes from other species. We created greater diversity in receptors through a complementary technique called error prone PCR. In this method, mutant receptors are created when their genes are replicated under suboptimal conditions where incorrect nucleotides are incorporated at random intervals. This second method of creating genetic diversity can be used in conjunction with gene shuffling to produce a larger number of variants and speed up the process of directed evolution.

Our accomplishments for FY 2005 are summarized below.

(1) We cloned the gerA operon from *Bacillus subtilis*.

(2) We developed a spore germination assay suitable for high throughput screening of germination mutants.

(3) We obtained a strain of *Bacillus subtilis* that contains a deletion of the gerA operon and tested this mutant with our germination assay.

(4) We are currently in the process of creating our first variant receptors by error prone PCR and by gene shuffling.

# Acoustic Sensors for Cell Signaling Proteins

*84314*

*S. M. Brozik, D. L. Huber, W. G. Yelton, D. W. Branch*

## Abstract

We proposed to extend our current research efforts in acoustic biosensors for live agent detection to the detection of viral particles and proteins involved in a specific cell signaling pathway. Long term, we will develop an integrated microfluidic system that will be used to isolate, identify, and track in real time specific proteins in the cell signaling cascade. Within the scope of the proposed work, we focused on development of the detection scheme. Two sensors were investigated for biosensing applications.

Specifically, we used the shear horizontal surface acoustic wave (SH-SAW) device to detect protein and viral particles in collaboration with the University of Texas system. In the past, the use of acoustic biosensors was applied only toward bacterial detection. Our preliminary results suggest promise of the SH-SAW device working for viral detection as well as for protein detection. We also developed a set of computer codes to permit analysis and screening of waveguide materials for predicting optimum mass sensitivity to reduce the number of experiments needed to determine optimum waveguide thickness.

We also evaluated the use of electrochemical patterning of thiolated B-cyclodextrin derivatives on electrode arrays. For the detection of multiple proteins involved in cell signaling pathways or in detection of multiple viral analytes, it is important to be able to pattern different capture molecules such as antibodies or peptides onto the array.

## Accomplishments

- SH-SAW Sensor:

(1) Optimize the performance of the SH-SAW sensor for viral and protein detection.

We developed several Love-wave configurations that use both delay-line and resonant structures. Our experimental results show that higher frequency devices can achieve detection limits into the pg/cm$^2$ regime, as compared to the 103 MHz Love-wave sensors (e.g., ng/cm$^2$). In this case, we compared detection limits of two Love-wave sensors on $LiTaO_3$ by capturing bovine serum albumin (BSA) using whole anti-BSA antibodies attached to the sensing surface. Sequential injections of BSA showed that the 325 MHz sensor detected 400 pg/cm$^2$, where the lower limit of the 103 MHz sensor was about 5 ng/cm$^2$.

For the detection of live agents, our previous result for detection of *Bacillus* spores was ~800 spores/ml. In this study, we began optimization of the sensor for viral agents. We were able to detect ~1,000,000 viral particles. This work led to follow-on funding for a project in which our goal is to continue improvement in the surface chemistry and waveguide material to enable detection limits of 10,000–100,000 viral particles.

(2) Investigate optimal waveguide materials:

Our current efforts focus on developing new tools to design optimized Love-wave sensors. These tools involve the use of computer simulation to aid in waveguide design and optimization. We developed a set of computer codes to permit analysis and screening of waveguide materials for predicting optimum mass sensitivity. Prior to this development, optimization was entirely empirical, requiring a large number of experiments for each parameter change. We are further extending this work by developing a model to address acoustic loss in the waveguide, an effect that has not been previously addressed in the literature. This model will also serve to understand the impact of altering the viscoelastic properties of the waveguide for sensitivity enhancement.

- Electrochemical Sensors:

(1) Investigate chemistries in which selective recognition layers can be produced.

We succeeded in patterning three different thiolated B-cyclodextrin derivatives to gold electrodes. The cyclodextrin film will then serve as a template for the reversible electrochemical patterning of surfaces with ferrocene and ferrocene derivatives. Initial results suggest that ferrocene is absorbed into the ring structure. We are investigating selective absorption, as well as release of the molecules from the cyclodextrin rings through oxidation and reduction of Fe(II) and Fe(III). In the future, the ferrocene molecules will be functionalized with antibodies or peptides and tailored to adsorb and desorb from cyclodextrin films deposited on gold electrodes. Focus of this study centered around the synthesis and electrochemical switching of the

three cyclodextrin films, all involving the technique of cyclic voltammetry using ferrocene, ferrocene carboxylic acid (FCA), and ferricyanide in a 0.2 M KCl supporting electrolyte.

With solutions of ferrocene, FCA, and ferricyanide, we were able to produce excellent responses from the oxidation and reduction of the iron couple on bare gold, platinum, or carbon electrodes. These experiments verified that the cyclic voltammetry protocol was an appropriate technique and was sensitive to the redox reaction of ferrocene and FCA in water. Upon coating a bare gold electrode, the thiolated B-cyclodextrin derivative (MJF-59) did not allow facile access of the ferrocene complex to the gold surface, yielding poor current response. On the other hand, the ferricyanide quickly entered the film but was nearly impossible to remove.

The ethylene glycol terminated B-cyclodextrin derivative (MJF-103) deposited on bare gold did not allow electron transfer between the modified electrode and any of the solutions.

Finally, di-alkyl sulfide B-cyclodextrin derivative (MJF-69) films deposited on gold performed well, allowing electron transfer to the surface upon oxidation/reduction of ferrocene, FCA, and ferricyanide. Continued work in this area will focus on (1) developing the chemistry to functionalize ferrocene or some other mediator to interact with a biological molecule and (2) controlling the distribution of the mediator in the cyclodextrin film.

# Thermal Accommodation of Carbon Nanotube Coatings

*84316*

*D. J. Rader, W. M. Trott, J. N. Castaneda, T. W. Grasser, J. R. Torczynski*

## Abstract

The purpose of this project was to explore the accommodation of thermal energy during collisions between gas molecules and a metal surface coated with carbon nanotubes. On one hand, a nanotube coating offers the possibility of a high degree of accommodation because of its fractal-like morphology. This porous structure would trap incident molecules so that they would undergo many gas-solid collisions before being reflected back into the gas in near-thermal-equilibrium with the solid. On the other hand, a discontinuous nanotube structure might act as an insulator so that the molecules are reflected with a temperature much less than that of the underlying metal substrate. It is the microscopic structure of the nanotube coating that will ultimately determine the macroscopic heat-exchange properties of the surface. The goal of this work is to experimentally investigate the relationship between the details of the nanotube coating and the resulting global thermal accommodation. Ultimately, this work would be of interest to a wide range of engineering problems. For example, nanotube coatings might be considered as a method for controlling heat transfer across microelectromechanical system (MEMS) micro channels or in the design of thermally insulating coatings for high-speed aircraft.

## Accomplishments

Heat transfer to surfaces immersed in noncontinuum (rarefied or transitional) gas flow continues to be an active area of research. Gases exhibit noncontinuum effects when the characteristic system length scale becomes comparable to the gas mean free path. These effects become important when either the system length scale becomes small (e.g., microelectromechanical systems) or when the gas pressure becomes low (e.g., semiconductor manufacturing or spacecraft aerodynamics). In these cases, a priori prediction of noncontinuum gas phase heat flux requires a detailed description of the gas-surface interaction. Unfortunately, and despite considerable effort over the past century, reliable gas-surface interaction mechanisms are still lacking. It is well recognized that the remedy to this dilemma lies in the development of a reliable, experimentally validated database of gas-surface interaction models.

The goal of this project was to experimentally measure the effect of surface roughness on gas-surface thermal accommodation. The thermal accommodation coefficients were to be determined using a previously developed experimental facility located at Sandia. In this facility, measurements are made of the heat transfer between two 6 inch diameter parallel plates separated by a small (~1 cm) gas gap. Accommodation coefficients are inferred from the measured dependence of the heat flux on gas pressure.

The base material selected was 304 stainless steel, a commonly used metal. Researchers studied three distinct surface preparations using 304 stainless steel: (1) a mirror polished finish (a smooth surface), (2) a standard machined (lathed) surface (a moderately rough surface), and (3) a carbon nanotube coated surface (a fully rough surface). The original plan was that the carbon nanotube coating would be performed at the University of Texas at Dallas (UT Dallas). Researchers shipped two stainless steel plates to UT Dallas for this purpose, but these plates have not been coated due to the difficulty in uniformly coating such a large area. UT Dallas staff are continuing to work on overcoming this challenge, and the nanotube coated plates will be tested when received.

Researchers completed thermal accommodation measurements for the polished and machined 304 stainless steel plates in contact with three gases: helium, nitrogen, and argon. They measured surface roughness of the polished plates (~20 nm) to be a factor of 100 less than that of the machined plates (~2 microns). Despite this large difference in surface roughness, they were unable to measure any detectable difference in the thermal accommodation coefficients for nitrogen (80% diffuse reflection) and argon (87% diffuse reflection). The accommodation coefficient for helium with the machined plates (36%) was slightly less than that measured for helium with the polished plates (40%). Based on these measurements, it appears that the surface roughness of 304 stainless steel has a very small effect on thermal accommodation.

## Refereed

W.M. Trott, D.J. Rader, J.R. Torczynski, J.N. Castaneda, and T.W. Grasser, “Measurements of Thermal Accommodation Coefficients,” presented at DSMC 2005: Theory, Methods, and Applications, Santa Fe, NM, September 2005.

## Other Communications

D.J. Rader, W.M. Trott, J.R. Torczynski, J.N. Castaneda, and T.W. Grasser, “Measurements of Thermal Accommodation Coefficients,” Sandia Report SAND-2005-6084, Albuquerque, NM, 2005.

# Sensor for Remote Detection and Monitoring of Uranium Enrichment and Conversion Processes

*86360*

*D. R. Wheeler, T. W. Hamilton, M. W. Moorman, L. F. Anderson, J. M. Anderson, S. W. Howell*

## Abstract

This report summarizes our team's initial development of a novel microfabricated sensor system to detect the elevated HF levels found in the environment around the production or enrichment of $UF_6$. Our system consists of a microelectromechanical system-based capacitive sensor with a precision deposited sensing film and a temperature control circuit to control humidity on the film. The use of microsystem components results in a reduced power requirement and system size, allowing it to perform as a remote and unattended sensor. We tested the sensor over a variety of conditions and operated it by capacitance monitoring of an oxide film that is removed by HF. Initial results indicate that sensor temperature control results in removal of excess humidity from the sensing film, which increased the HF reaction rate with the $SiO_2$ film. We demonstrated HF detection in the low ppm to high ppb concentrations.

## Accomplishments

Extensive development work took place to provide proof-of-principle for this sensor concept and to begin development of an HF sensing system. We fabricated and tested two generations of microsensor devices, investigated four different sensing film deposition methodologies, and produced a temperature control circuit. Sensor testing occurred over a variety of relative humidity levels, temperatures, and flow rates and demonstrated HF detection in the low ppm to high ppb concentrations. Results show that HF has a strong reactivity with $SiO_2$, which is the primary component of this film, and results in the removal of the $SiO_2$. The removal of this film occurs regardless of the sensor's power status and can be measured as a shift in capacitance. This gives the system capability to operate in an extremely low power integrator mode to establish the presence of HF over the long term. The variety of sensing film deposition techniques we experimented with further allows us to tailor the sensor's etching duration and the capacitance shift caused by the film. Temperature modification, using the temperature control circuit, proved to affect signal level by driving adsorbed moisture from the sensing film and speeding the etch rate, according to initial indication.

*Image of sensor on a US dime.*

## Other Communications

L. Anderson, S. Howell, R. Manginell, M. Moorman, and D. Wheeler, "Sensor for Remote Detection and Monitoring of Uranium Enrichment and Conversion Processes," presented to NA22 at the Department of Energy, Washington, DC, 2005.

# Terahertz Spectral Signatures: Measurement and Detection

*86361*

*M. Lee, M. C. Wanke, C. Highstrete, I. Brener, E. A. Shaner, J. L. Reno*

## Abstract

Terahertz (THz) spectra have a singular advantage over microwaves and infrared in their ability to identify many molecules and solids from their spectral resonances with very high specificity. Thus, one of the most widely sought after applications of THz spectroscopy is the remote, or stand-off, detection and identification of various chemical molecules and materials of interest. The goal of this project was to increase Sandia's capabilities to measure THz signatures and to begin using that knowledge in designing an integrated, matched, and tuned solid-state remote detection subsystem.

We significantly enhanced the THz spectroscopy infrastructure. We designed and constructed a picosecond pulsed THz time domain spectrometer. This system is meant to be a user friendly, flexible spectrometer capable of measuring transmission and reflection spectra in the frequency range from approximately 0.2 to 3 THz for a wide variety of materials in both vapor and solid phases. The system demonstrated signal-to-noise of better than 40 dB. Also, we added a reflectometer setup and additional vapor phase gas cells to an existing THz Fourier transform spectrometer (FTS), greatly increasing its capabilities.

We collected several example THz signatures from various chemicals and solids using these systems. These include the absorption spectra of water, carbon monoxide, methanol, and nitrous oxide, all in vapor phase, and the reflection and transmission spectra of zinc oxide and strontium titanate in solid phase.

We achieved a major milestone toward designing a tuned solid-state transceiver with the design, fabrication, and successful testing of a quantum cascade laser operating at 2.9 THz. The frequency and power parameters of such a laser are necessary input parameters in designing a THz receiver system matched to a spectroscopic signature line.

## Accomplishments

We designed and built a THz time domain spectrometer (THz-TDS) that generates picoseconds and, hence, broad spectral bandwidth pulses from sub-picosecond infrared pulses using an electro-optic ZnTe crystal. By varying the time delay between pulse generation and detection, the THz-TDS measures a time domain profile of the THz electric field. A digital Fourier transform conversion yields spectral information.

The THz-TDS unambiguously determines both spectral amplitude and phase, allowing direct determination of complex optical properties such as index of refraction. The system covers a spectral range of about 0.2 to 3 THz with spectral resolution of a few GHz. The spectrometer also achieved a signal-to-noise ratio of about 40 dB, giving it great dynamic range. We designed this system to be flexible enough to accommodate the interests of potential customers interested in THz spectra.

We also upgraded a THz ultra-high resolution FTS by adding reflectometry capability and extending the number of gas cells that can be used for vapor phase absorption spectroscopy. This instrument is unusual compared to most Fourier spectrometers in that its frequency coverage goes down to 0.15 THz and its spectral resolution is exceptionally fine, $< 0.002\ cm^{-1}$. The very high resolution of the FTS is important, both to establish intrinsic properties of a molecule and to delineate any closely spaced lines in a spectrum that may serve as a narrow band distinguishing signature. For high altitude or exoatmospheric applications, having a sharp spectral line database is necessary because spectra are not pressure broadened under such conditions.

We extensively tested both spectroscopic instruments by acquiring sample THz signatures for a number of molecules and materials. These include the THz absorption spectra of carbon monoxide, methanol, nitrous oxide, and water, all in vapor, and the reflectance and transmission spectra of silver, gold, zinc oxide, and strontium titanate in solid form.

Such signature knowledge ties together the ongoing solid-state THz source and detector projects in many conceivable end user systems. For example, in high performance sensing applications, a narrow band, quantitatively known spectral signature defines the frequency and sensitivity performance demands around which a tuned, matched source/detector heterodyne receiver system must be designed and built. The best example is the NASA Aura satellite launched July

2004, which carries a tuned OH remote sensor system consisting of a Schottky diode mixer with a fixed-frequency molecular gas tube laser as its local oscillator (LO), all tuned to a known 2.5 THz signature of the hydroxyl radical (OH). With this decade old technology, this system measures tropospheric OH concentrations using sunlight as the illumination source. Our ultimate goal is to construct a similar transceiver system that replaces the large, finicky, expensive gas laser with a microelectronic quantum cascade laser (QCL) as the LO. We achieved a major milestone toward making this work with the successful design, fabrication, and testing of a QCL operating at 2.9 THz. The performance of this device is competitive with state-of-the-art research in such devices, and we estimated that it can provide sufficient LO power to a Schottky diode mixer to operate the mixer near its minimum noise point.

## Other Communications

E.A. Shaner, M.C. Wanke, M. Lee, A. Grine, J.L. Reno, and S.J. Allen, “Plasmon Based Grating Gate THz Detectors,” presented at EP2DS-16, Albuquerque, NM, July 2005.

# PCSS/Fiber-Optic Trigger System for Pulsed Power Switches

*86362*

*K. W. Reed, F. J. Zutavern, S. F. Glover*

## Abstract

The purpose of this project is to develop fiber-optically triggered photoconductive semiconductor switches (PCSSs) to trigger high voltage switches with improved precision and eliminate the need for large diameter trigger cables or line-of-sight optics. The goal of this phase of the work is to determine PCSS design criteria, switching properties, and trade-offs for a PCSS based trigger system for the direct current (DC) charged Russian linear transformer driver (LTD) switch, utilizing an intermediary switch. During FY 2005, researchers addressed this trigger development in three stages:

(1) Testing a PCSS based trigger generator triggering a 100 kV DC charged trigatron intermediary switch;

(2) Triggering an existing LTD brick with a Russian switch using the PCSS based trigger circuit and intermediary switch;

(3) Optimization of the trigger circuit.

## Accomplishments

- *First Milestone:* The PCSS trigger system was initially tested independently in a 55 ohm load, representing the resistance that will be in series with the trigatron trigger pin. The PCSS controlled the discharge of a 180 pF capacitor that was pulse charged in a series of experiments to 14 kV, 15 kV, 16 kV, and 17 kV. Measurements with a load current viewing resistor (CVR) and an output D-dot probe showed that these charge voltages yielded an 11 kV, 12 kV, 13 kV, and 14 kV forward going voltage pulse that is 8 ns wide with a 2 ns rise time. We reduced the peak pulse voltage from the charge voltage by the on-state switch voltage (~1.5 kV) and the system inductance, which limits the rise time and peak current of the capacitive discharge.

The measured root mean square timing jitter from the laser light pulse on the PCSS to the current pulse in the load was 298 ps. This result was a success. Doubling at the trigatron trigger pin in the next experiment, these results will yield trigger voltages of 22 kV to 28 kV, spanning the trigatron design value of 25 kV. Most pulsed power trigger jitter requirements are several nanoseconds or more.

The goal of the next step was to demonstrate triggering of the trigatron. We connected the trigger system through series 50 ohm resistors to the trigger pin of the trigatron intermediary switch. The trigatron discharged a 667 pf + 67 pf capacitor that was DC charged to 100 kV into a 28 ohm load. Measurements included the laser light pulse on the surface of the PCSS, the voltage and current into the trigger pin of the trigatron, and the current in the trigatron load. Results indicate that we were able to trigger the trigatron successfully, and the current in the load is in agreement with the predicted waveform based on a 700 pf capacitor.

- *Second Milestone:* The PCSS pulse generator and trigatron were added to an LTD test bed with a Russian +/-100 kV switch. The PCSS/trigatron trigger system was used to trigger the Russian switch in a configuration that replicated an LTD brick. The PCSS successfully triggered the trigatron, and, in turn, the trigatron successfully triggered the Russian brick. Current from the Russian switch and previous diagnostics on the PCSS trigger generator were used to confirm successful triggering.

- *Third Milestone:* Optimization of the PCSS/trigatron trigger system is under way. Redundant diagnostics from each of the subsystems (PCSS pulse generator, the trigatron, and the Russian switch) were calibrated and compared to each other and to the expected waveforms. We can determine the minimum current, voltage, and pulse widths required for reliable, low jitter triggering by adjusting subsystem charging voltages, capacitances, and impedances. We are monitoring and reducing internal reflections, which may limit the lifetime of the PCSS by changing component values, improving subsystem impedance matches, and reducing stray inductances and capacitances.

## Other Communications

F.J. Zutavern, K.W. Reed, S.F. Glover, A. Mar, M.H. Ruebush, M.L. Horry, M.E. Swalby, J.A. Alexander, and T.L. Smith, “Fiber-Optic Controlled PCSS Triggers for High Voltage Pulsed Power Switches,” presented and in *Proceedings of the IEEE 2005 Pulsed Power Conference*, Monterey, CA, 2005.

# Exploiting Interfacial Water Properties for Desalination and Purification Applications

*90493*

*R. T. Cygan, P. J. Feibelman, B. C. Bunker, C. J. Brinker, T. M. Nenoff*

## Abstract

Water is the critical natural resource of the new century. Significant improvements in traditional water treatment processes will require novel approaches based on a fundamental understanding of nanoscale and atomic interactions at interfaces between aqueous solution and materials. Active research tasks are examining the fundamental behavior of interfacial water, with emphasis on two-dimensional (2-D) surfaces and 3-D confined water systems. This project will: (1) identify those properties of interfacial water whose better scientific understanding could lead to new desalination/purification technology opportunities; (2) define the new experimental, theoretical, and simulation approaches to predictive understanding of these key properties; and (3) provide a plan that links research successes in these areas to follow on technology opportunities. Our expertise in surface science and chemistry, theory and simulation, in situ characterization, and the synthesis of well-defined porous materials and fluidic architectures will enable Sandia to succeed in this science-based approach to engineering improved desalination membranes, urgently needed to alleviate the worldwide potable water supply crisis.

To better understand these critical issues and to promote an open dialogue among leading international experts in water-related specialties, we sponsored a workshop on April 24–26, 2005, in Santa Fe, New Mexico. The “Frontiers of Interfacial Water Research Workshop” provided attendees with a critical review of water technologies and emphasized the new advances in surface and interfacial microscopy, spectroscopy, diffraction, and computer simulation needed for the development of new materials for water treatment.

## Accomplishments

The first efforts of the project aim at gaining knowledge of current related work to develop our research strategy, and for initial demonstrations of the methods, we are using to obtain key information on interfacial water.

To better understand critical issues and to promote an open dialogue among leading international experts in water related specialties, we sponsored a workshop in April 2005, in Santa Fe, New Mexico. The “Frontiers of Interfacial Water Research Workshop” provided attendees with a critical review of water technologies and emphasized the new advances in surface and interfacial microscopy, spectroscopy, diffraction, and computer simulation needed for the development of new materials for water treatment.

The international workshop helped us identify outside collaborators, and we identified mutual efforts with the University of Illinois Center of Advanced Materials for Purification of Water with Systems and with Ilja Siepmann at the University of Minnesota. The workshop pinpointed key results for analysis: “ice-like” layers are not seen in spectroscopic and diffraction examinations of water-oxide systems, notwithstanding earlier claims in the literature. The workshop also highlighted related areas for research, such as how antifreeze proteins function, how ice freezes to exclude solution electrolytes, the structure of water on mineral surfaces in the environment, and state-of-the-art methods in the simulation of interfacial water processes.

The research strategy we implemented is divided into the study of 2-D and 3-D environments. Our 2-D work takes advantage of the ability to “see” what is happening at a water interface with a flat high quality surface. It also allows us to use a high degree of control in a “surface science” environment to isolate the surface properties (geometric and chemical) that control interfacial water structure and transport. By applying a wide range of analytical techniques, we have a reasonable chance of discovering the nature of what we know to be a very complicated interfacial environment. We are initially examining the behavior of water on the well defined and atomically flat mica surface as a basis for much of the characterization and theoretical analyses.

There are two distinct threads to the 3-D efforts. One is using Sandia expertise in synthesis to fabricate model pores of desired size, shape, and surface functionality. This allows us to test concepts for improved purification strategies in quasi-realistic experiments. The other thread involves using classes of ion-

exchange materials whose properties can be varied systematically to establish structure-property relationships directly. In both cases, theorists working closely with our experimenters are simulating and parameterizing the experimental results, thereby supporting the data interpretation and helping to develop new concepts for the development of new materials for water treatment.

## Refereed

R.T. Cygan and J.A. Greathouse, "Frontiers of Interfacial Water Research: Workshop Report," Sandia Report SAND2005-6220, Albuquerque, NM, 2005.

P.J. Feibelman, "Stress Correction for Slab Asymmetry in Supercell Calculations," to be published in *Physical Review B.*

# Binary Electrokinetic Separation of Target DNA from Background DNA Primers

*91312*
*C. D. James*

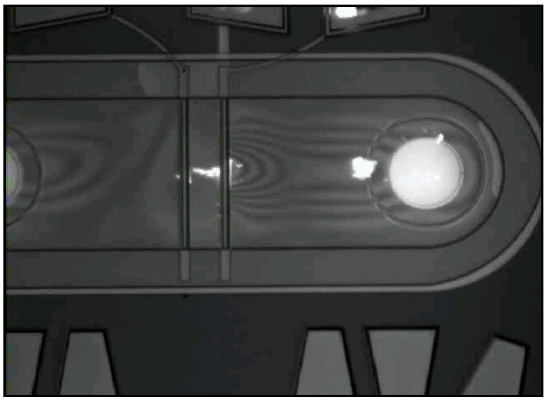
## Abstract

Researchers at Columbia University were the first to identify the West Nile virus and, more recently, the SARS (severe acute respiratory syndrome) virus. They found these organisms using the digital strand diagnostic display broadband polymerase chain reaction (PCR) technique that they pioneered. They recently created a panel of assays for the list of hemorrhagic fevers using their unique mass tag technology.

We are developing a technique to reduce false positive events, due to the detection of unhybridized reporter molecules, in a sensitive and multiplexed detection scheme for nucleic acids developed by the Lipkin laboratory at Columbia. This is the most significant problem in the operation of their capability. As they are developing the tools for rapidly detecting the entire panel of hemorrhagic fevers, this technology will immediately serve an important national need. We are collaborating with Columbia and the Northeast BioDefense Center of Excellence.

The goal of this work was to attempt to separate nucleic acid from a preprocessed sample. We demonstrated the preconcentration of kilo length double-stranded deoxyribonucleic acid (DNA) targets and observed little preconcentration of 60 length single-stranded DNA probes. We accomplished these objectives in microdevice formats that are compatible with larger detection systems for sample preprocessing.

Combined with Columbia's expertise, this technology would enable a unique, fast, and potentially compact method for detecting and identifying genetically modified organisms and multiplexed rapid nucleic acid identification. Another approach is the Defense Advanced Research Projects Agency funded IRIS Pharmaceutical TIGER platform, which requires many hours and an $800K piece of equipment that fills a room. The Columbia/Sandia system could provide a result in 30 minutes, cost a few thousand dollars for the platform, and would be the size of a shoebox or smaller. Further development of the work demonstrated in this proposal will overcome a major hurdle in miniaturizing this biodetection system into a portable moderate cost platform.



*Video clip of release of preconcentrated dsDNA from a DEP gate. To collect dsDNA, the gate is set to 20 V p-p, 1 MHz. The voltage is set to 0 V to release the band of DNA.*

## Accomplishments

We demonstrated the selective preconcentration of kilobase-pair double-stranded DNA (dsDNA). Using 20 V p-p 1 MHz voltages, we were able to preconcentrate dsDNA into pre-defined regions. Trapping was fast (time constant of ~2 seconds) and, depending on the device configuration, reversible. Small single-stranded oligonucleotides were not preconcentrated and can, thus, be flushed from a flow-through system. Two different device configurations were used: (1) an interdigitated electrode chip and (2) a surface micromachined microfluidic flow-through device. The interdigitated electrode device contained a large number of traps; in this device, a considerable portion of the trap region exists in regions between microelectrodes. This promoted reversible trapping of dsDNA that released the DNA upon turning off the voltage. The flow-through microfluidic device contained only one trap, but the integrated fluid channel permits continued and controlled delivery of DNA to the trap region. A drawback to this device lies in the fact that the bulk of the trapping region is directly at the microelectrode edges, leading to some level of irreversible binding of trapped DNA to the electrodes. A combination of the design elements of each device would be optimum for high throughput reversible DNA preconcentration and binary large target, small probe separation.

# Nanostructured Surfaces for Microfluidics and Sensing Applications

*91991*

*J. A. Voigt, N. Bell, M. Peich, J. Houston, J. Taraci, S. Ingole, D. Yang*

## Abstract

The objective of this research is to develop new understanding of the nanoscale structure dependence of surface hydrophobicity and to exploit new optically controlled methods of changing the hydrophobicity for manipulating microliter to nanoliter quantities of fluids on surfaces. Such understanding would enable development of new approaches to lab-on-a-chip materials synthesis and chemical/biochemical analysis. The research conducted during this project consisted of synthesizing nanostructured surfaces, developing monolayer photoswitchable coating methodologies, and measuring the wetting properties of these hydrophobic surfaces. We conducted the research in collaboration with Arizona State University.

Three major new accomplishments were achieved:

(1) we demonstrated photoswitchable surface hydrophobicity on ZnO nanostructured surfaces for the first time using a spiropyran monolayer design;

(2) we developed a new photoswitchable monolayer system based on a tethered azobenzene design, and these surfaces show the largest water contact angle optical switching we have achieved to date on smooth surfaces;

(3) we demonstrated the first measurements of the force to move air bubbles on superhydrophobic silicon nanowire surfaces, providing a microscopic non-optical method to investigate surface contact angle hysteresis.

## Accomplishments

(1) *ZnO surfaces:* We carried out extensive studies to develop the new surface chemistry processes required to coat ZnO nanopillar surfaces with spiropyran-based monolayers. Well controlled ZnO surfaces with different nanopillar diameters, spacing, and shape have been developed at Sandia. We previously demonstrated, on monolayer coated superhydrophobic silicon nanowire surfaces, that large amplification of photoswitching could be achieved simultaneously with significant decreases in the contact angle hysteresis for water. This enabled the first demonstration of the movement of water droplets using only light (to change the surface state) and thus opened the possibility of the control and manipulation of microliter to nanoliter sized water droplets on surfaces for application in new approaches to lab-on-a-chip sensing, analytical chemical analysis, and nanoscale surface synthesis.

We initially encountered significant issues with the chemical preparation method previously used for silicon oxide surfaces for the ZnO nanopillar surfaces. The sensitivity of the ZnO to pH levels below ~6 resulted in a chemical attack of the ZnO nanostructures, and byproduct formation during the process steps led to a contaminant layer. We developed changes in the surface process to greatly reduce these problems. A more reproducible photoactive monolayer design containing spiropyran is now available. Reversible photoswitching of the water contact angle between 130 and 152 degrees for ultraviolet and visible light exposure was achieved on ZnO nanopillar surfaces. This key accomplishment demonstrated the switched high and low polar surface states and the ability to optically control surface hydrophobicity on ZnO.

(2) *Azobenzene photoswitchable coating:* The surface sensitivity of ZnO motivated us to search for new monolayer photoswitchable surface coatings that would be robust and simpler to implement. As a result, we developed a new photoactive coating design based on tethering azobenzene to the surface. This new process required only two steps (versus three for the spiropyran monolayer design). On smooth surfaces we demonstrated 15 degree reversible photoswitching, the largest seen to date. Because of the different nature of the photochemical switching process between azobenzene and spiropyran, we now have a new tool for understanding photoswitchable hydrophobicity on nanostructured surfaces.

(3) *Understanding surface bubble motion:* We measured the force to move air bubbles (under water) on surfaces and discovered a new non-optical method to determine the contact angle hysteresis, using an interface force microscope. We found that when a hydrophobic tip is brought near our superhydrophobic silicon nanowire surfaces, a micron sized air bubble is spontaneously formed. By analyzing the video recorded change in advancing and receding contact angles simultaneously with the measured force to move

the air bubble across the surface, we found a well defined behavior. The force and the hysteresis in the contact angles increase simultaneously until bubble motion starts, at which point the values remain constant. This process is the inverse of moving a water droplet across the surface but provides a much more reproducible and quantitative approach. These two independent measures of surface energy change are directly correlated and agree well. This process can be used to map the contact angle hysteresis on surfaces and to understand the local changes in hysteresis with surface morphology and chemistry. The hysteresis in surface wetting (one can think of this as the stickiness of the surface) is perhaps one of the oldest and least understood aspects of fluid interactions with surfaces, yet it is perhaps the most critical parameter in controlling fluid droplet interactions with surfaces. In earlier work, we discovered that contact angle hysteresis can be strongly reduced by the addition of nanoscale surface morphology. We now have a method to interrogate this change as we vary morphology and seek to understand the change in wetting hysteresis across tailored surfaces.

## Other Communications

S.T. Picraux, J.L. Taraci, D. Yang, P. Aella, M. McLauchlin, M.D. Gust, A.A. Garcia, and M. Hayes, “Tailored Inorganic/Organic Nanostructured Interfaces for Control of Surface Hydrophobicity,” presented at the Fall Materials Research Society Meeting, Boston, MA, November 2005.

D. Yang, “Optical Control of Hydrophobicity Through Surface Nanostructure and Monolayer Coatings,” presented at the Research in Engineering and Applied Science Annual Meeting, Arizona State University, September 2005.

# Assessment of Quantum Dot Photoluminescence Measurement Techniques

*94124*

*G. A. Samara, E. D. Jones*

## Abstract

Sandia recently completed an LDRD project entitled "Nanocrystal-Polymer Composites for High Luminous Efficiency LEDs" and reported on resulting progress made in creating nanoparticle-based phosphors. However, major differences in the reported luminescence results, as much as a factor of two, were observed using two different measuring techniques.

The purposes of this project are to evaluate the two experimental techniques, to make determinations regarding any fundamental differences between the two methods for determining phosphor quantum yields, and to determine whether or not errors were made during previous analyses of the data. Specifically, the major goals are:

(1) to compare quantum yield data and reproducibility from the two different techniques;

(2) to assess some claims that Sandia researchers were the first to:

(a) observe decoupling of the absorption and photoluminescence in quantum dots;

(b) produce broadband emitting nanophosphors based on simple binary nanocrystals (or quantum dots of CdS/CdSe); and

(c) use surface active molecules or surfactants to restructure the cluster surface leading to broadband white light emission from a single size nanocluster of CdS.

## Accomplishments

Our assessment is that the two different types of photoluminescence measurement techniques yield very similar results, i.e., the same values for the quantum yield are found from both kinds of measurements. However, one of the experimental measurements apparently suffered from errors in the analysis of the data, yielding incorrect results. Our conclusion is that it is likely that significant errors were made by using rhodamine 6G as a reference standard and not accounting for the effects of self absorption. These errors were propagated by calibrating coumarine 500 in methyl alcohol as a secondary standard against rhodamine 6G. Subsequently, measuring quantum yields of the broadband (white light) emitting quantum dot samples, with respect to coumarine 500 in methyl alcohol quantum yields, led to errors of as much as a factor of two.

A more extensive examination of past experimental results on quantum dot luminescence, as reported in the literature, was performed. We found early studies by other researchers who made significant contributions to the field of quantum dot broadband emitters, including a patent awarded for a broadband quantum dot CdS LED.

# Atomic Layer Deposition of Boron Nitride Films for Microsystems Lubrication Applications

*94235*
*R. K. Grubbs*

## Abstract

Hexagonal boron nitride, h-BN, is an ideal solid lubricant for microsystem applications. Its hexagonal crystal phase has a low coefficient of friction in air at 0.15, and it is electrically insulating, thermally conductive, and chemically stable. Successful BN coatings could lubricate the mechanical parts of a microelectromechanical system (MEMS) without short-circuiting the electrical components. Its chemical stability and thermal conductivity also makes BN well suited for microsystems. What is needed is a deposition technique that can chemically coat hidden, shadowed, or non-line-of-sight areas, e.g., gear hubs, with a thin and conformal lubricious film.

The focus of this project was to utilize the atomic layer deposition (ALD) process for BN chemistry using non-halogenated precursors. We performed initial experiments on a silicon substrate using the halogenated precursor, $BCl_3$, as this was easier to acquire and much less expensive than $B(CH_3)_3$. The initial BN growth experiments showed that amorphous BN thin films could be deposited in a flow reactor modified to accept static doses. A thin BN film was shown to exist on the substrate by depth profile auger electron spectroscopy. X-ray diffraction (XRD) measurements of the BN films suggested a strong amorphous character to the ALD grown films. We per-formed preliminary annealing studies on these films and observed no conversion to either hexagonal or cubic crystal structure when annealed up to 800 °C for 5 minutes. An increased temperature anneal of 1100 °C for 5 minutes created a single phase highly crystalline boron silicate. Using the knowledge gained using $BCl_3$, further experiments using the non-halogenated boron precursor would be insightful.

## Accomplishments

We set out to determine the feasibility of performing BN deposition by ALD in a flow reactor using nonhalogenated precursors. It was first important to modify the present ALD reactor to accommodate the increased chemical exposures necessary for the BN ALD chemistry. We modified the reactor (and software) to run in a quasi-static mode where the chamber could become a static dosing chamber as well as a classic ALD flow reactor. This modification provided the large exposures necessary for BN growth without the loss of expensive precursors. The first films grown were difficult to detect. Optically transparent and with no x-ray signature, it was hard to prove that any thin film had grown. We attempted to modify the substrate surface to increase the probability of nucleation by exposing it to a $H_2O$ plasma or growing it on ALD $Al_2O_3$ surfaces. These modifications would have added a -OH on the surface to become the nucleation handle for the BN chemistry.

We performed auger depth profiles measurements on some of the samples and determined the presence of BN, along with C and O impurities. The depth profile showed that the film was predominately BN. Further depth profiling revealed the underlying $Al_2O_3$ substrate. This was confirmation that BN ALD chemistry was working. When we analyzed samples grown at various temperatures (300–475 °C) by x-ray, there was no hint of structure, either hexagonal or cubic, that indicated that these films were amorphous.

We performed annealing studies on the ALD grown BN films. Film annealed at 700 °C and 800 °C for 5 minutes showed no change in the x-ray pattern. Another attempt heated a sample to 1100 °C for 5 minutes. This anneal produced a very single phase boron silicate that was confirmed via x-ray and auger depth profiling.

It appears that BN films can be grown in a modified ALD flow reactor. The limited annealing studies were unable to witness a conversion from amorphous to hexagonal crystal structure at modest Si processing temperatures.

It should be noted that we obtained the above results using the halogenated precursor $BCl_3$ rather than the proposed non-halogenated precursor $B(CH_3)_3$. Due to the cost of $B(CH_3)_3$ and the delivery time, it was fruitful to use $BCl_3$ to provide preliminary results on the chemistry of ALD of BN. Further research will need to be performed using the proposed $B(CH_3)_3$.

# Mission Technologies

The Mission Technologies investments aim to create or accelerate the technical innovations that provide solutions to difficult national security problems. Investments in Mission Technologies also seek to extend the boundaries of the science and technology into fertile new areas that will support current and future missions.

The focus of Mission Technologies investments falls into four investment areas: Energy and Infrastructure Assurance, Military Technologies and Applications, Nonproliferation and Assessments, and Homeland Security.

**Energy and Infrastructure Assurance** investments support Sandia's national security mission by developing and applying technologies in three primary thrust areas: energy surety, global nuclear future, and water. While work across these thrust areas is primarily focused on technical challenges within the US, a number of these challenges have fundamentally global dimensions. Projects focus on new concepts, ideas, and technologies needed to increase the sustainability (availability and utilization) of domestic energy supplies, to reduce our dependence on imported energy, and to minimize the impact of energy production and utilization on the environment and natural resources. Energy and Infrastructure Assurance endeavors also work to improve the security and reliability of our energy infrastructures.

**52539 "A Combined Preconcentrator and Sensor for Live Water Borne Pathogens"**

Researcher Eric Cummings examines an insulative dielectrophoresis (iDEP) prototype that is used to separate live from dead water borne pathogens.

For example, project **52539 "A Combined Preconcentrator and Sensor for Live Water Borne Pathogens,"** combines research efforts in chemical and earth sciences, sensing and detection, and infrastructure assurance. The goal of the project, discussed in the Chemical and Earth Sciences area, is to develop a method to dielectrophoretically allow timely analysis of the safety of water supplies. Researchers developed an insulator-based electrodeless dieletrophoretic concentrator that uses applied direct current fields to catch and release particles from fluid streams. Results demonstrate selectivity in sorting live, dead, and bacterial species; effectiveness of injection-molded plastic devices; and 6000X concentration per stage. This will be the first device that uses physics that occur at the microfluidics scale to take large sample volumes to small sample volumes. Work thus far resulted in several publications in peer reviewed journals, one filed patent, and another in preparation. The project addresses a critical gap in all proposed rapid bacterial, viral, spore, and protozoan analysis methods needed for selective concentration of the microbes of interest. This gap must be bridged to protect critical infrastructure, including water utilities.

**79820 "Portable Medical Diagnostic System for Detection of Presymptomatic Biomarkers of Chem/Bioagent Exposure"**

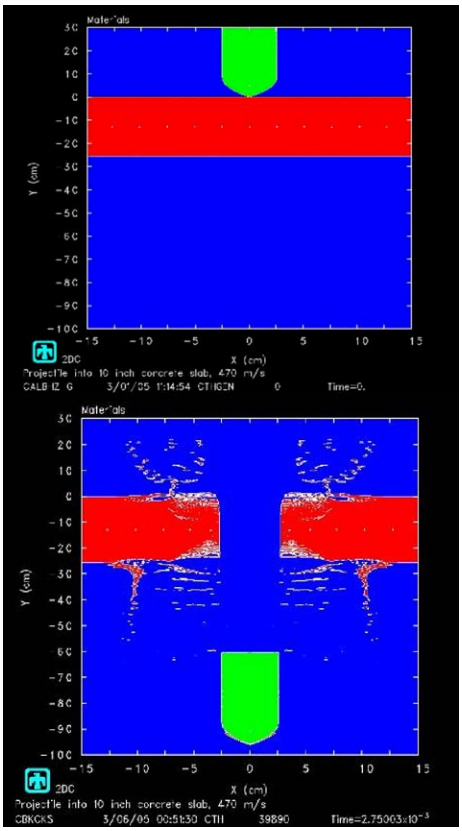
Researcher Anup Singh inserts an immunoassay chip into the microfluidic module of the hand-held device.

**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.

Project **79820 "Portable Medical Diagnostic System for Detection of Presymptomatic Biomarkers of Chem/Bioagent Exposure"** is working to develop a portable device for rapid screening and triaging of a population after a suspected incident of bioterrorism. The device will be capable of carrying out multianalyte presymptomatic detection in minutes using a tiny amount of blood or saliva. It will also be able to detect definitive and specific biomarkers such as surface antigens, toxins, or antibodies. The device is based on microfluidic technologies that manipulate and concentrate biomolecules to perform selective recognition of analytes using immunoassays. In a proof of concept demonstration, researchers were able to detect toxins similar to anthrax in a matter of minutes compared to the hours required for conventional methods of detection. This work addresses a critical need in responding to potential bioterrorism attacks: a rapid and accurate method of positively identifying those citizens exposed to a bioagent before symptoms are visible and while there is still time to begin treatment. Many of the problems and solutions Homeland Security investments undertake overlap other national security mission areas; for this reason, projects are strongly encouraged to create multidisciplinary teams that cross cut the research community.

The mission of the **Military Technologies and Applications** investments is to strengthen national security by anticipating and delivering high-impact solutions to a broad range of defense challenges. This is accomplished in large part through the application of the advancement and development of 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. Research in these areas includes new directed-energy weapon and interrogation systems, decision and reasoning aids, threat detection, target characterization, hypersonic vehicles, intelligent systems, modeling and simulation, threat mitigation, communication and tracking systems, systems analysis, miniaturization of sensors, laser countermeasure systems, remote sensing, and sensor systems.

More specifically, project **79837 "Non-Inertial Electric Field Void Sensor"** investigates the feasibility of using alternative non-inertial sensors for buried target defeat that do not rely on projectile acceleration to sense whether the projectile is in a void (air space). The sensors would also be able to develop a history of the voids they



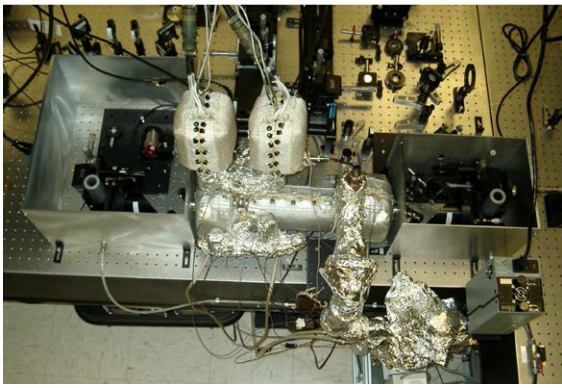

### 79837 "Non-Inertial Electric Field Void Sensor"

Data from modeling a debris field of a 14 inch long penetrator passing through a 10 inch thick concrete wall shows the penetrator outran the fragments. The effect of the debris field on the interpretation of the sensor's signals is encouraging.

encounter. Current accelerometers for penetrator weapons are not very reliable, and detonation may not occur at the optimum location. This type of sensor provides a new paradigm to achieve fuzing for optimal effect. The sensors can be made small, and some of their subsystems demonstrate high-g tolerance. Results indicate excellent target discrimination capability. Researchers investigated concrete, sand, water, and black soil versus air. Researchers modeled a penetrator debris field to determine its affect on the sensor's signals interpretation. The penetrator outran the fragments; this was an encouraging result for interpretation of the void sensor return signals. The project indicates that the sensors could be a very valuable tool for optimizing the effectiveness of penetrating munitions.

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 aim to 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 efforts in weapon detection and characterization; radiation detection; geolocation; engineering solutions for smaller, lighter, lower power systems; information collection; advanced algorithms for analysis, modeling, and simulation; information operations; next generation tamper indicating devices; monitoring systems; biological detection and risk assessment; physical security; sensing, detection, and assessment architectures; and ultraviolet, radio frequency, electro-optical, and next generation sensor concepts.

For instance, project **67111 "Atmospheric Propagation of THz Radiation"** seeks to develop accurate characterization of atmospheric contaminants at various altitudes using terahertz (THz) radiation. This type of characterization will help determine



### 67111 "Atmospheric Propagation of THz Radiation"

The Terahertz time-domain spectrometer used to study the propagation of THz radiation through the atmosphere measured the absorption spectrum of atmospheric water vapor at varying relative humidities. The spectral range of the measurement extend from 0.6 to 2.6 THz with a spectral resolution of 8 GHz.

atmospheric transmission (signal attenuation is a major problem) for remote sensing of explosives, biological and chemical agents, and cold-body tracking. Researchers built an operational gas sample cell that they can use to not only conduct THz measurements of atmospheric gas samples, but also to conduct THz spectral measurements of any solid, liquid, or gas materials of interest. The cell is rated to conduct experiments at both ambient and high temperatures. Researchers also improved the performance of the THz spectrometer and detection system. Net signals in the time domain went from microvolts to millivolts where the detection limit of the THz spectrometer is 3 ppm.

# Energy and Infrastructure Assurance

## Beyond Nanoparticles—Attack on a Chemical "Holy Grail"

*52591*
*R. A. Kemp, J. E. Miller, C. A. Stewart*

### Abstract

The ability to selectively oxidize organic molecules directly using oxygen gas is one of the "Holy Grails" in catalytic chemistry. This ability is known as such due to the huge energy savings possible if organic molecules can be converted, directly and efficiently, into other reactive molecules and intermediates. While a simple olefin like ethylene can be epoxidized selectively to ethylene oxide using heterogeneous silver catalysts, higher olefins such as propylene and cyclohexene containing allylic hydrogen atoms cannot be epoxidized at all using the same technology. These epoxides currently must be produced via multi-step synthetic routes. In this study, we are examining the production of higher olefin epoxides (oxiranes) using molecular oxygen as the oxidizing species and homogeneous, late transition metal pincer complexes as possible catalysts. We hypothesized that a homogeneous catalytic cycle, beginning with a metal hydride, can react with oxygen to form a metal hydroperoxide. This hydroperoxide can donate an oxygen atom to generate the epoxide, and after reaction, a metal-hydroxide species is formed. The rest of the proposed catalytic cycle involves regenerating the metal hydride by using either carbon monoxide or hydrogen as a transforming agent. In order to fundamentally understand this cycle better, we examined each proposed step in the cycle individually in stoichiometric reactions. We individually demonstrated each step of this catalytic cycle in isolation and developed a single system (a phosphorus-pincer palladium complex) that can perform each of the steps.

### Accomplishments

During the course of this project, we examined a large number of different organometallic ligand-metal combinations to attempt the direct conversion of higher olefins (such as propylene or cyclohexene) to their respective epoxides using molecular oxygen as the oxidant. If we are successful, this breakthrough technology can affect the study of all partial oxidation processes. While we have not solved this problem, we made tremendous progress toward understanding and developing a homogeneous route to these epoxides. We believe that transition metals that appear early in the periodic chart are too oxophilic to complete the catalytic cycle. Therefore, we concentrated our efforts on middle and late transition metal complexes.

We spent a great deal of effort attempting to use chelating, N,O-donating salen ligands with various metals, such as iron and cobalt. However, the complexes proved difficult to characterize and cleanly identify. Concurrent with this, we were also examining N- or P-ligated pincer ligands to Pt and Pd. With a single PCP-pincer ligand complexed to Pd, we individually demonstrated all the steps of the proposed catalytic cycle. We also characterized some of the intermediates explicitly using x-ray crystallography. Using the PCP-Pd metal ligand combination, we synthesized and definitively characterized the (PCP)Pd-H complex, believed to be the key intermediate in our catalyst cycle. We demonstrated the insertion of oxygen gas into this species to form (PCP)Pd-OOH, which we identified by its x-ray structure. This is the first exam-

ple of a well characterized insertion of oxygen into a Pd-H bond.

Further experiments demonstrated that the insertion of oxygen was not a radical process. The (PCP)Pd-OOH complex could donate an oxygen atom to some acceptors more cleanly than others. Reaction with propylene (the main target molecule) gave a mixture of products, but reaction with cyclohexene gave cyclohexene cleanly. Reaction with triphenylphosphine gave the triphenylphosphine oxide cleanly. After oxygen atom transfer, a (PCP)Pd-OH species remained. The species could be converted back to the (PCP)Pd-H starting material by reaction with carbon monoxide to form a well characterized metal hydroxycarbonyl followed by decarboxylation. We also discovered a new route to regenerate the desired (PCP)Pd-H complex by using hydrogen gas, then eliminating water from the presumed (PCP)Pd$(H)_2$-OH intermediate for the Pd-H species. Thus, we can mimic nature by capitalizing on the elimination of water to regenerate our catalyst.

We also examined a number of other P-pincer-metal combinations and pincers that contain other heteroatoms, such as N or S. These were not as successful as the P-ligated pincers in our studies. We attempted to prepare new fluorinated pincer ligands for use with metal complexes in perfluorinated solvents. As the solubility of oxygen is significantly higher in fluorinated solvents versus hydrocarbon solvents, we hoped that the increase in available oxygen in these systems would lead to more selective reactions. However, the synthetic chemistry to prepare these ligands without cleavage of the tails is difficult. The status at the end of the project for the perfluorinated solvents is less certain than that with the PCP-ligands.

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# Advanced Proton-Exchange Materials for Energy Efficient Fuel Cells

*52592*

*C. J. Cornelius, G. S. Grest, D. Ingersoll, C. L. Staiger, M. Hibbs*

## Abstract

The purpose of this project was to initiate the fundamental research necessary for the development of a novel proton-exchange membrane (PEM) to overcome the material and performance limitations of the state-of-the-art Nafion used in both hydrogen and methanol fuel cells. An atomistic modeling effort was added to the project to establish a framework between predicted morphology and observed PEM morphology related to fuel cell performance. Significant progress was made in the area of PEM material design, development, and demonstration. A fundamental understanding was developed about the role of the structure of the PEM material as a function of sulfonic acid content, polymer topology, chemical composition, molecular weight, and electrode electrolyte ink development in improving fuel cell performance. PEM materials based upon random and block polyimides, polybenzimidazoles, and polyphenylenes were created and evaluated for improvements in proton conductivity, reduced swelling, reduced $O_2$ and $H_2$ permeability, and increased thermal stability. Results from this work reveal that the family of polyphenylenes potentially solves several technical challenges associated with obtaining a high temperature PEM membrane. Fuel cell relevant properties such as high proton conductivity (> 120 mS/cm), good thermal stability, and mechanical robustness were also demonstrated.

## Accomplishments

A fundamental understanding was developed that related the structure of the PEM material, the content of sulfonic acid, molecular weight, polymer morphology, chemical composition, and electrode electrolyte ink to improve fuel cell performance. PEM materials based upon random and block polyimides, polybenzimidazoles, and polyphenylenes were created and evaluated for improvements in proton conductivity, reduced swelling, reduced $O_2$ and $H_2$ permeability, and increased thermal stability.

Results from this work reveal that the family of polyphenylenes potentially solves several of the technical challenges associated with obtaining a high temperature PEM membrane. The polyphenylenes, synthesized via Diels-Alder condensation, resulted in polyelectrolytes that could be post-sulfonated in a controllable manner with chlorosulfonic acid. The aryl backbone resulted in a tough rigid-rod material, indicated by its high glass transitional temperature ($T_g$) and inherently high thermochemical stability. This type of high $T_g$ material possesses physical properties that are attractive in high temperature fuel cell applications. In addition, the backbone stiffness does not negatively affect membrane properties such as water uptake (21 to 137%) and proton conductivity (13 to 123 mS/cm) with ion exchange capacity that are typically employed with sulfonated aromatic polymers (0.98 to 2.2 meq/g). The resultant polymers could be solution cast into robust, creaseable films. They also displayed properties that suggested this class of sulfonated polymer is a potential candidate for hydrogen and methanol fuel cell applications. We are exploring strategies to enhance their conductivity especially under partially dry conditions. This work led to direct funding from industry and from the Department of Energy/Energy Efficiency and Renewable Energy fuel cell program for further development.

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# Real-Time Discriminatory Sensors for Water Contamination Events

*52595*

*C. D. Mowry, R. P. Manginell, W. Einfeld, D. R. Wheeler, S. M. Dirk, D. E. Trudell, K. S. Rawlinson, D. R. Adkins, J. L. Barnett, S. K. Showalter, A. Robinson, T. W. Hamilton, W. C. Chambers, A. M. Goodin, G. J. Shelmidine, D. Barfoot, K. Carrejo Simpkins, A. N. Irwin, J. A. Ellison, T. T. Borek III, P. C. Fletcher, R. Hadizadeh, R. T. Miner*

## Abstract

The goal of this project was to apply Sandia's surface acoustic wave (SAW) array detector technology to the detection of volatile trihalomethanes (THMs) in water, and we achieved this goal. Another goal met was demonstration of the applicability of Sandia microfabricated devices to issues of collection, separation, and detection of additional analytes, like those found in chemical weapons. This project had an objective to perform a field test at a water utility with a portable system. While a portable system was assembled, this objective was not met.

THMs are byproducts of disinfection processes followed by water utilities. Levels of THMs are regulated by the Environmental Protection Agency (EPA) due to their threats to human health. Current methods for THM analysis are costly, slow, and used primarily for the imposition of fines rather than for the protection of consumers. Rapid, on-site detection is not available for THMs at this time, yet the EPA is in the process of implementing more stringent monitoring and lower regulatory levels. For public safety, mitigation steps need to be performed immediately, rather than days or weeks after sample collection and subsequent detection of contaminates or high THMs.

At the beginning of FY 2005, we identified preliminary components for collection, separation, and detection, but we had not yet fully tested them. We designed and built field system electronics (acquisition, control, and power distribution) but did not fully test or integrate them.

At the end of the fiscal year, we assembled a complete working field system and qualified individual components. The accomplishments in the next section are presented in each of the following categories: system, collection (preconcentration), separation, detection, electronics, and fluidics. We achieved an operable field system. However, we did not achieve the final objective of a field test. Many difficulties with preconcentrator technologies (building, fluidics, and performance) delayed the schedule. Ultimately, fluidic issues and detection limits of currently available SAW detector coatings prohibited a relevant field test.

## Accomplishments

The fully operational and self-contained field system follows the procedure of purge-and-trap for collection and desorption, gas chromatography for separation, and detection using SAW detection. Completion of the system was the most significant accomplishment in FY 2005. The accomplishments enabling an operational system are discussed below in the categories of system, collection (preconcentration), separation, detection, and fluidics.

- *System*: We assembled and tested a functional field system for the detection of THMs in water. The system is robust for travel, includes real-time data plotting and post processing, and weighs only 32 pounds. Contained in a carry-on luggage style case of dimensions 22 by 13 by 7 inches, it will run continuously for at least two hours. One can collect a grab sample, turn the power on, then the push of a single button performs the analysis and stores the data. Significant miniaturization is still possible through improved packaging. We also built a second water sampling reservoir that will enable on-line monitoring.
- *Collection (preconcentration):* Testing determined that previously designed and built preconcentrators (ohmic bead) were not reliable. A new design was implemented, and new materials were tested for suitability to the humid gas flow and volatile analytes. In addition, we performed purging efficiency studies to validate the assumption that field efficiency (air purge) would be similar to laboratory efficiency (helium purge). We determined a decrease of approximately 50% with air purge, suggesting lower detection levels were needed for the SAW detector.
- *Separation:* We fully qualified a column phase type previously identified as a potential candidate for field use (resolution, robustness, rapid separation) under field conditions and a variety of flow conditions. We verified field temperature control parameters against a laboratory oven. We assembled spare field

columns for possible field replacement needs. We packed microfabricated columns with candidate materials and tested them for THM separation. The biggest advantage of the columns is the ability to temperature ramp with low power. One packed column provided excellent separation results.

- *Detection:* We tested a variety of SAW detector coatings for efficacy towards THM detection. A Sandia designed polymer designated as "DKAP" (a fluorinated phenol siloxane polymer) provided reversible response at room temperature but not at the levels required for field monitoring at EPA-regulated levels. We tested an alternative and designated it as "PPP" (polyphenylated phenylate). However, there was not enough time or budget to optimize the coating technique for this polymer to achieve suitable results to warrant a field test. While this polymer showed enhanced response, the reversibility was also slow, broadening peaks. Two possible untested solutions would be higher temperature SAW operation and/or further coating optimization.
- *Electronics:* We tested and integrated previously designed electronics boards this year. We debugged software and modified it to allow for full control of flow, heating, timing, flow valves, desorption, sensing, and data collection and output by an inexpensive, commercially available programmable integrated circuit. We created a calibration method and tailored it for field data using a commercial software package. In addition, we wrote a LabView program to display data in real time during field data collection. We identified a noise problem and proposed a solution.
- *Fluidics:* The previously designed field system fluidics were problematic with new preconcentrator devices, so we redesigned and remachined them. We tested the new fluidic block and installed it into the field system.

## Other Communications

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# Advanced High Efficiency Direct Cycle Gas Power Conversion Systems for Small Special Purpose Nuclear Power Reactors

*52596*

*S. A. Wright, P. H. Helmick, E. J. Parma Jr., P. S. Pickard, G. E. Naranjo, M. E. Vernon*

## Abstract

Small nuclear power systems offer enabling technology for new and challenging Department of Energy (DOE), National Aeronautics and Space Administration (NASA), and Department of Defense (DoD) commercial space and terrestrial applications. Directly coupling high temperature gas turbine cycles to gas cooled nuclear reactors has the potential to dramatically increase cycle efficiency up to 35% for space applications and up to 50% for terrestrial applications. Such significant efficiency improvements would enable next generation missions such as next generation high temperature gas reactors, nuclear electric propulsion and power for space systems, and specialized advanced DoD missions.

The purpose of this project was to study and validate the coupling of nuclear reactors to gas dynamic Brayton power conversion systems. The research focused on developing integrated dynamic system models, fabricating a 10–30 kWe closed-loop Brayton cycle, and validating these models by operating the Brayton test loop. Operation of the test loop and development of the system models has allowed Sandia to develop and validate a set of tools/models that can be used to determine how nuclear reactors operate with gas turbine power conversion systems. These tools are proving useful for developing and evaluating control strategies and for modeling even larger reactor systems such as high temperature gas reactors and other next generation systems.

We performed tasks in three major areas. First, we developed the system equations and dynamic models for reactors and closed Brayton cycle systems and published them in several reports. Second, we modified an open-cycle, commercially available Capstone C30 microturbine power generator to provide a small inexpensive closed Brayton cycle test loop. We operated the test loop at the manufacturer's site (Barber-Nichols Corporation) and installed and operated it at Sandia during the summer of 2005. Third, we operated the test loop to develop a more complete understanding of this electrically heated closed power generation system and to validate the dynamic model.

## Accomplishments

We achieved accomplishments in three major areas.

First, we developed and implemented the system equations and dynamic models for reactors and closed Brayton cycle systems in a dynamic state flow simulation programming language, Simulink™, and published results in several reports and conferences. Within this effort, we developed and assembled both steady state and dynamic system models for all the components (turbines, compressors, reactors, ducting, alternators, heat exchangers, and space based radiators) into complete systems for gas cooled reactors, liquid metal reactors, and electrically heated simulators. The simulation package is called RPCSIM (Reactor Power and Control SIMulator). We used this dynamic model to study the control behavior of reactors and to develop operational behavior maps and representative start up transients for reactor driven closed Brayton power conversion systems.

Second, we completed the development of a 10–30 kWe Brayton cycle test loop and control system. We operated the system and compared it to the dynamic system model (RPCSIM). We performed this effort by modifying a commercially available open-cycle Capstone C30 microturbine power generator. We operated the test loop at the manufacturer's site (Barber-Nichols Corporation) to develop steady state and dynamic data. Later in the year, we installed the system and operated it at Sandia. We developed a sufficiently detailed description of the Sandia Brayton test loop (SBL-30) that, when used in conjunction with the characteristic flow maps of the turbo-alternator-compressor, permits the researchers to compare their results with those measured in the Sandia test loop.

Third, we operated the test loop to develop a more complete understanding of the electrically heated closed-loop power generation system and to validate the dynamic models. Tests are currently ongoing to assess the effect that gas conductivity, molecular weight, and gas pressure have on the test loop. We are performing other tests to assess the capability of the Brayton engine to remove decay heat after the reactor/heater is shut down, to develop safe and effective con-

trol strategies, and to assess the effectiveness of gas inventory control as an alternative means to provide load following.

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# Novel Catalysts for Hydrogen Fuel Cell Applications

*52598*

*S. M. Thornberg, R. L. Jarek, J. E. Miller, E. N. Coker, W. A. Steen*

## Abstract

The primary objective of this project was to develop novel hydrogen-oxidation electrocatalyst materials for fuel cell applications that contain reduced platinum content. Our approach was to (1) apply fundamental materials science (sophisticated experimental characterization and thermodynamic/kinetic modeling) to identify optimum nanocluster chemical and physical catalyst configurations, (2) develop flexible synthesis techniques to fabricate these configurations on supported structures, and (3) measure and verify electrochemical performance ultimately in laboratory fuel cells.

This project resulted in the development of a novel procedure for synthesizing Pt/C electrocatalysts that allow unprecedented control over Pt cluster size and catalyst nanostructure. The procedure involves the following: (1) in situ formation and stabilization of size controlled Pt clusters within a microporous zeolite material, (2) introduction of a secondary carbonaceous matrix via impregnation and in situ decomposition processes, (3) conversion of the carbonaceous matrix into conductive (graphitic) carbon, and (4) final digestion of the zeolite host to produce the novel Pt/C catalyst structure. Pt clusters with as low as sub-nanometer dimensions and with desired narrow size distributions have been stabilized on the zeolite host.

To assist the development and validate performance improvements, a suite of half-cell electrochemical experiments were performed on the Pt/C materials. Metrics included electroactive surface area and, ultimately, catalytic activity. Electroactive surface areas were similar to, and in many cases exceeded, those for benchmark commercial Pt electrocatalysts, indicating that reduced Pt loading could be achieved in fuel cells. The most challenging requirement for any electrocatalyst involves its effectiveness in the oxygen reduction electrode. In a laboratory fuel cell, our Pt/C material performed admirably with voltage losses approximately the same as the best catalysts (~400 mV). Furthermore, relative to commercial benchmark materials, we have evidence that our materials are more durable, and a staggering 50% more power was produced in an extended 300-hour test.

## Accomplishments

The primary objective of this project was to develop novel hydrogen-oxidation electrocatalyst materials containing reduced platinum content for fuel cell applications. Our approach was to (1) apply fundamental materials science (sophisticated experimental characterization and thermodynamic/kinetic modeling) to identify optimum nanocluster chemical and physical catalyst configurations, (2) develop flexible synthesis techniques to fabricate these configurations on supported structures, and (3) measure and verify electrochemical performance ultimately in laboratory fuel cells.

We developed a novel procedure for synthesizing Pt/C electrocatalysts that allows unprecedented control over Pt cluster size and catalyst nanostructure. The procedure involves: (1) in situ formation and stabilization of size-controlled Pt clusters within a microporous zeolite material, (2) introduction of a secondary carbonaceous matrix via impregnation and in situ decomposition processes, (3) conversion of the carbonaceous matrix into conductive (graphitic) carbon, and (4) final digestion of the zeolite host to leave the novel Pt/C catalyst.

Pt clusters, down to sub-nanometer dimensions and with narrow size distributions, were stabilized in the zeolite host. Key variables in achieving the size control include the concentration of Pt in the zeolite and the rate at which the sample is heated during cluster formation. To form a carbon matrix with good electrical conductivity, carbon precursor-impregnated samples must be heated to a minimum of 800 °C. The presence of the carbon matrix reduces agglomeration of the Pt clusters during this high temperature treatment, compared to identical treatment of a Pt zeolite without secondary carbonaceous matrix. The final electrocatalysts possess Pt clusters with sizes tunable from ~1.5–4 nm and with Pt loadings of between 5 and 40 wt%. One notable aspect of the ability to tune cluster size is that fundamental electrochemical properties can now be evaluated. Under optimized synthesis conditions, Pt/C catalysts show enhanced Pt surface areas compared to commercial benchmark materials, and ordered (zeo-

litic) microporosity of the carbon matrix can be achieved.

To assist the development and validate improved catalyst performance, we performed a suite of half-cell electrochemical experiments on the Pt/C materials. Metrics included electroactive surface area and, ultimately, catalytic activity. Electroactive surface areas were similar to, and in many cases exceeded, those for benchmark commercial Pt electrocatalysts, indicating that reduced Pt loading could be achieved in fuel cells. Reducing the Pt requirement was one of the overarching goals of this effort, and we can boast a successful outcome. For example, one of our 40 wt% Pt/C materials had 112 $m^2$ per gram Pt, compared to a commercially available 20 wt% Pt/C material having 86 $m^2$ per gram Pt. Increased electroactive surface area is certainly insufficient to achieve success in that the material must also serve as a kinetically facile electrocatalyst.

The most difficult reaction, and therefore the most challenging requirement on any electrocatalyst, is to minimize the potential drop associated with the reduction of oxygen and also provide a long service life. By collaborating with other Sandia fuel cell related projects, several catalyst performance characterization measurements were made in actual laboratory fuel cells. First, our Pt/C material showed real promise with voltage losses on the oxygen electrode, approximately the same as the best commercial catalysts (~400 mV). Furthermore, long term catalyst stability is an area of active research and concern within the fuel cell community. We have evidence that the unique design of our materials may indeed enhance service life and long term catalyst stability. Finally, and of real significance, our material produced 50% more power than a similar, commercially available catalyst in a 300-hour test.

## Refereed

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# Linking Optimization and Simulation in Critical Infrastructure Systems

*52606*

*D. A. Jones, M. A. Turnquist, C. A. Phillips, L. K. Nozick, C. E. Davis*

## Abstract

This research focused on the development of modeling tools for understanding the performance of complex infrastructure systems, assessing their vulnerability, and guiding investments to improve the security of these systems. Three key features have distinguished this work over the course of the project. The first is a focus on networks (graphs of nodes and arcs) as a common modeling architecture to represent systems of interest. The second is the complementary use of simulation and optimization in the modeling tools developed. Third, we have focused considerable attention on the potential for malicious intruders to do damage to the systems of interest and on strategies to protect the systems from such intrusions.

In this third year, our primary focus was on a model of malicious intrusions in infrastructure facilities, using a network representation of the system structure together with Markov models of intruder progress and strategy. This structure provides an explicit mechanism to estimate the probability of successful breaches of physical security and to evaluate potential improvements. Simulation is used to analyze varying levels of imperfect information on the part of the intruders in planning their attacks. An example of an intruder attempting to place an explosive device on an airplane at an airport gate illustrates the structure and potential application of the model.

## Accomplishments

The objective of this project is to develop models for understanding the performance of complex infrastructure systems, assessing their vulnerability, and guiding investments to improve the security of these systems. A cornerstone of this project is network based modeling. Networks (graphs of nodes and arcs) can represent capacities of components in infrastructure systems and their evolution over time, the condition of supporting information system components, the location, and condition of an intruder in a system, etc. Network based models also represent significant opportunities to integrate simulation and optimization.

During the final year of this project, our primary objective was to be able to provide guidance to system owners and operators regarding effective ways to reduce vulnerabilities of specific infrastructure facilities. To accomplish this, we developed a Markov decision process (MDP) model of how an intruder might try to penetrate the various barriers designed to protect the facility. The solution to this MDP model provides insight into the level of vulnerability of the facility (the probability of successful intrusion) and indicates where the vulnerabilities are (the most likely paths for the intruder).

Lower level models of intruder detection at barriers (nodes) of the system can be built as hidden Markov models, and the results of those lower level models can be aggregated for use in the MDP of intruder strategy for attacking the system. A key aspect of this analysis is representing imperfect information on the part of the intruders. Simulation is used as a tool to evaluate the effects of varying levels of imperfect information, sampling from distributions of detection probabilities, and using those samples to construct distributions of intruder path choices through the network and overall success probability.

A small example problem illustrates that even relatively small amounts of uncertainty in the information the intruders have about the system can significantly affect the probability that they can mount a successful attack. If the uncertainty is combined with bias in the perceptions of some system parameters, the effect on the intruders is magnified. In the small example studied, biased perceptions of two key detection probabilities combined with small amounts of uncertainty in perceptions of all the detection probabilities reduce the likelihood of a successful attack by a factor of about two. In addition to allowing us to estimate the probability of a successful intrusion, the simulation also allows us to estimate the likelihood of attacks appearing at specific locations in the network. This is very useful information for security forces.

The intruder model also provides the basis for consideration of possible strategies to reduce the probability of a successful attack on the facility. Illustrations of using the model in this way are provided in a case study analysis. The process of searching for cost-effective strategies to reduce system vulnerability can be

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# Agent-Based Control of Distributed Infrastructure Resources

*64811*

*L. R. Phillips, M. A. Cook, A. A. Akhil, R. B. Smith, H. E. Link, K. L. Stamber, J. E. Stamp, L. A. Weiland*

## Abstract

The goal of this project is to exhibit the benefits of using a distributed agent coalition to operate a distributed infrastructure. We also want to demonstrate the feasibility of such a system by exhibiting agents based on our Sandia developed standard agent architecture performing relevant operations with respect to a simulated electric power microgrid.

Distributed systems tend to be more efficient and robust and fail more gracefully than centrally controlled systems. Today, distributed infrastructures are usually operated and managed by humans using telephones to coordinate their actions. These operators receive data about system conditions and issue system commands by direct interaction and via supervisory control and data acquisition (SCADA) systems. This ordinarily works reasonably well but can break down catastrophically under stress. For the electric power system, broad distribution would require tens of thousands of new small generators. Such an arrangement is unlikely given the difficulty of operating that many elements in a coordinated fashion, even with modern SCADA systems. A distributed infrastructure management mechanism that can quickly resolve local issues and coordinate regional response is needed.

Our approach is to manage the infrastructure using autonomous distributed agents that maintain normal operation, enforce operational and security policy, deal with contingencies, and protect against errors, malicious insiders, and cyber attacks. We specify a distributed agent coalition that is able to accomplish this with distributed electric power, exhibit the design of the individual agents, specify the coalitions they need to form and the communications required, and describe a prototype implementation based on Sandia developed technology.

## Accomplishments

The primary contribution of our research is the design of an agent-based grid-control framework that details the agent operations needed to operate a power grid. We developed and operated a proof-of-principle demonstration in which the agent coalition interacts with a human operator to exhibit the feasibility of the concept.

The agent-based control of distributed infrastructure resources (ABCDIR) system manages the utilization of electric power generation and distribution components. Agents monitor voltages and power flows, recognize and respond to contingencies, and accept and carry out requests from human operators. They take action when the current state indicates a reconfiguration of the power components is necessary to achieve liveness conditions or avoid violation of safety conditions and upon receipt of authorized commands.

In steady state, agents periodically sample local sensor data to populate the local elements of an internal model of the grid. When significant changes in local state occur, an agent shares this new state information with the other agents. ("Significant" change is a change in voltage or power magnitude beyond a threshold value for a duration that exceeds a threshold time.) Similar thresholds trigger action planning. For example, "The power flow through circuit breaker X indicates that branch 391 will be running at greater than 95% of its rated capacity. If this condition persists for 20 minutes, open the breaker at the branch's upstream end node to prevent line overload. Initiate planning to reduce the power flow to 90% or less."

Elements of the design include the following: (1) a list of liveness and safety conditions, (2) a description of the goals of the agent-based microgrid management system, (3) a detailed explanation of the roles that the agents need to be able to take on, (4) a description of the interactions between the roles and the protocols needed to complete the specified interactions, (5) descriptions of how the agents relate to the elements of the electric power system, and (6) descriptions of the organizations formed by the agents.

In order to complete the demonstration, we also developed the following items: (1) general and detailed scenario scripts that specify the desired agent behavior in some detail; (2) the flow estimation algorithm executed by the agents; (3) an alternating current power-flow simulation of the scenario microgrid, a partial instantiation of the agent coalition specified by our design; and (4) a proof-of-principle demonstration of agents executing a scenario of interest.

The agent-based microgrid management system design produced by this project resolves the primary issues of agent functionality for distributed infrastructures in general, and for microgrids in particular: What is the function of the agents in steady state and under contingency? What is the nature of their interactions with one another and with humans? By what means does the agent coalition provide security? and How do agents enforce both operations policy and security policy?

The results of the project provide a requirements analysis, a software system specification, an agent system design, and a proof-of-principle implementation. Sandia can now approach potential system users and collaborators with these elements in hand to engage in a process that will result in an implemented agent-operated microgrid. With additional effort, the same agent system management framework could be applied to other infrastructures such as natural gas, petroleum, water, logistics, supply chains, and communication.

# Biological Security Decontamination Technology—Reducing the Threat of Infectious Agent Spread

*67050*

*R. G. Betty, M. D. Tucker, J. M. Bieker*

## Abstract

Currently, there are no highly integrated response capabilities to outbreaks of highly infectious agricultural diseases. These diseases, either intentional or the result of a natural outbreak, are a major concern to the economic well being of the US. Through the combination of enhanced bio-security decontamination remediation and validated inactivation verification methodology, we intend to provide important technologies that could easily be incorporated as components of a robust response capability. Because of the potentially devastating economic impact that could result from the spread of infectious agricultural diseases, the proposed capability components will promote a more secure infrastructure and greater border security.

We continued development of biosecurity decontamination formulations that will reduce the threat of infectious agent spread to plants, animals, and humans via surfaces inherent to an agricultural environment. In addition to ensuring wide spectrum decontamination efficacy and low corrosivity, biodegradability issues were addressed.

Through partnership with Kansas State University (KSU), we continued development of rapid verification of loss of pathogenic inactivity (inactivation) methodology. This enhanced technology will enable rapid assessment of contamination following an intentional event and will also be extremely useful in routine assessment of agricultural environments.

The primary effort during the second year was progress toward a demonstration of both decontamination and viral inactivation detection technologies of Foot and Mouth Disease virus (FMDv) developed through this project. Utilizing the decontamination technology developed for enhanced detergency in the presence of heavily loaded organic environments, the field test will serve as both a test for the decontamination technology and a validation of the rapid viral inactivation methodology on actual live infectious agents. The demonstration will be held at the Department of Homeland Security's (DHS's) Plum Island facilities. The increased response capability will benefit agencies such as the United States Department of Agriculture (USDA), DHS, and Department of Defense (DoD).

## Accomplishments

The following objectives were met in FY 2005:

- Sandia Component:

(1) Corrosivity testing of metal surfaces (galvanized tin) exposed for 24 hour periods to Decon Foam 200 (DF200), Virkon S, and modified formulations: This work incorporated the use of electrical impedance spectroscopy measurements.

(2) Surface detergency methodology: Limited resources were spent investigating alternative surface detergency methods as a potential replacement for the surface reflectance methodology. The conclusion was to continue use of the industry-standard surface reflectance measurements to assess surface detergency. The nearest alternative method of contact angle measurement was not a viable alternative because stickiness of surfactants would affect goniometer test results.

(3) An alternate surfactant of high biodegradability, meeting the Federal Drug Administration's (FDA's) requirements for use on food surfaces, was selected for potential use in modified biosecurity decontamination formulations. Efficacy testing indicated this surfactant is slightly more effective in decontamination of spore forming bacteria than the current surfactant in DF200. Surface detergency results of modified formulations with this surfactant also demonstrated an increased soil removal capability, particularly from butyl rubber surfaces.

(4) An evaluation of maximum surfactant sorption onto soil demonstrated potential for enhanced soil sorption of the alternative surfactant noted above.

(5) The testing of modified formulations (efficacy of standard spore kill protocol) in the presence and absence of an organic load challenge progressed well at Sandia. This work also included the surface detergency assessment, as measured by surface reflectance.

- KSU Component:

In this research, we conducted three studies. In the first study, bovine corona virus (BCV) was used preliminarily as a respiratory model surrogate virus to

study virus inactivation. The BCV model was primarily used to study a rapid verification for loss of viral infectivity based on degradation of ribonucleic acid (RNA) following disinfectant treatment. In the second study, we conducted experiments to study the recovery of virus samples from environmental matrices including soil and fecal material. The third study was the use of bovine enterovirus 2 (BEV-2) as a surrogate to FMDv to study viral inactivation. We treated BEV-2 virus inoculum with an equal amount of test decontamination formulation for various exposure durations (1, 10, 20 minutes). Additionally, we challenged the decontamination formulation with 0, 10, 25, or 50% bovine fecal challenge (10% bovine feces weight/volume). We washed samples via ultracentrifugation (100K xg, 1 hour, 4 $^{o}$C) to remove disinfectant and reconstituted pellets in appropriate infectivity media. We then evaluated samples using tissue culture infective dose 50 (TCID50) or with reverse transcriptase polymerase chain reaction (RT-PCR) to evaluate post-treatment infectivity and intact RNA, respectively. We conducted three replications for each sample treatment.

# Consequence Management, Recovery, and Restoration After an Intentional Contamination Event

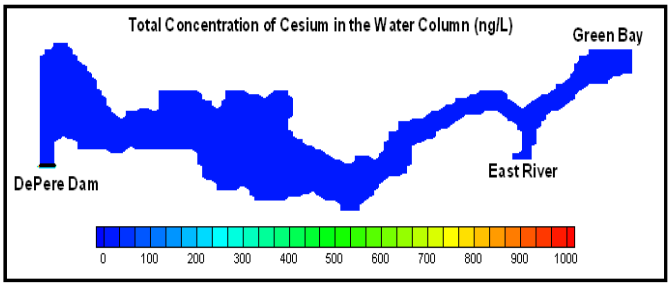
*67051*

*J. D. Roberts, S. C. James, V. C. Tidwell, D. Wall*


## Abstract

The fate of contaminants after a terrorist act is a major concern. Waterways are particularly vulnerable to contamination by such an attack. Contaminants could be introduced directly into a water system (municipal or general) or indirectly (radiological dispersal device) from aerial dispersion, precipitation, or improper clean-up techniques that may wash contamination into storm water drains, sewer systems, rivers, lakes, and reservoirs. Most radiological, chemical, and biological contaminants have an affinity for sediments and organic matter in the water system. If contaminated soils enter waterways, a plume of contaminated sediments could be left behind, subject to remobilization during the next storm or flood event, or contaminants could remain in place and damage local ecosystems for a long time. Suitable planning and deployment of resources to manage such a scenario could considerably mitigate the severity of the event. First responses must be pre-planned so that clean-up efforts do not increase dispersal and exacerbate the problem.

Interactions between the sediment, contaminant, and water cycle are exceedingly complex and poorly understood. This research focused on the development of a risk based model that predicts the fate of intentionally introduced contaminants in surface water systems. Achieving this goal requires integrating sediment transport with contaminant chemical reactions (sorption and desorption) and surface water hydrodynamics. Sandia leveraged its existing state-of-the-art capabilities in sediment transport measurement techniques, hydrochemistry, high performance computing, and performance assessment modeling in an effort to accomplish this task. In addition, the basis for the physical hydrodynamics is calculated with the Environmental Protection Agency (EPA) sponsored, public domain model, Environmental Fluid Dynamics Code (EFDC). The results of this effort will facilitate systems analysis and numerical simulation that allow the user to determine both short term and long term consequences of contamination of waterways. It will also help to formulate preventative and remedial strategies. Efforts in FY 2005 focused on a field demonstration of the model.


*Video clip of total concentration of cesium in the water column for the 5-year flood event and high partition coefficient.*

## Accomplishments

We met many of the milestones set for FY 2005 and modified others to streamline the research. Specifically, we met the following milestones:

(1) Developed and implemented sediment transport algorithms that resolve fine and coarse grained sediment transport processes.

(2) Developed and implemented algorithms to model kinetic reactions between sediment, contaminants, and water.

(3) Incorporated the input files with data from Sandia patented sediment erosion measurement devices.

(4) Demonstrated the model's utility to predict the spread of a hypothetical contamination event with a model of the Lower Fox River in Wisconsin.

(5) Demonstrated the model's ability to predict hydrodynamic circulation within a lake environment by modeling wind driven circulation within Cedar Lake in Indiana.

(6) Published one peer-reviewed journal article describing model process enhancements, authored two succeeding papers, and presented results at three professional conferences.

- Milestone that we modified:

(1) The Pecos River was not an appropriate site to demonstrate the model's full capabilities. We modeled alternate river and lake systems.

- Milestone not met:

(1) Due to time constraints, we did not run a full suite of stochastic model runs for sensitivity/uncertainty analysis. We did test the ability of Sandia's Alice computing cluster to run multiple realizations of this code. We identified and resolved weaknesses in the cluster organization and file management system.

- Lower Fox River Field Test:

We selected the Lower Fox River for field demonstration because it has been thoroughly studied and well characterized as a result of historical industrial contamination issues. The model introduces a pulse of highly hydrophobic contaminant (cesium) for six hours at its upstream boundary. The contaminant pulse was supplied at equilibrium between dissolved and particulate phases and comprised 10,000 g of cesium. We considered three scenarios:

(1) An average flow rate of 105 $m^3/s$ and average cesium partition coefficient (Kp = 1000).

(2) An average flow rate of 105 $m^3/s$ and high cesium partition coefficient (Kp = 10,000).

(3) A five-year flood event (425 $m^3/s$) and high cesium partition coefficient (Kp = 10,000).

For each scenario tested, significant localized areas of relatively high contamination remained within the model domain, while nearly the entire domain was contaminated to levels considered unacceptable by EPA standards. The average flow scenarios allowed more sediment to deposit, resulting in increased cesium residuals toward the river's edges. A high partition coefficient results in more cesium sorbed onto particulate material in the water column. Cesium associated with sediments may be transported or deposited depending on flow conditions.

- Cedar Lake Field Test:

Cedar Lake has multiple small inlets and one outlet at several locations along the shoreline. None of these tributaries represent a major hydrodynamic forcing on the lake. In the absence of anthropogenic factors, wind driven circulation and wave action are the primary factors governing the hydrodynamic behavior of the lake. Wind speeds during storm events are sustained at up to 17 knots.

A 17-knot north wind establishes a net movement of water at the lake surface toward the south. The velocities are largest near the shorelines in shallow water. At the mid-water column and near the bottom in the central lake region, there are return flows to the north that balance the southward flow of water at the surface. Additionally, there are downward velocities along the south shore and upward velocities along the north shore. Vertical circulation is also present at the narrow central portion of the lake. The modeled circulation, due to wind across the lake, is characteristic of observed wind-driven circulation on lakes.

For each wind direction, the highest lake bottom shear stresses are seen along the downwind shoreline of the lake where the combined wave heights and current speeds are the largest. The maximum shear stresses generated in the lake exceed 0.2 Pa. Preliminary sediment analyses indicate that surficial critical shear stresses of the sediments are between 0.05–0.10 Pa, indicating that sediment resuspension and contaminant recycling could be initiated in several areas of the lake.

## Refereed

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# Fully Integrated System Dynamics Toolbox for Water Resources Planning

*67052*

*V. C. Tidwell, O. H. Paananen, H. D. Passell, L. A. Malczynski, J. L. Krumhansl*

## Abstract

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 the myriad of values that influence water policy. We are working to create a modular and generic resource-planning "toolbox" that would lead to such a tool.

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, 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 the integrated modeling of water resource systems. This toolbox will also provide the technical foundation and framework for creating an interactive, icon driven system dynamics modeling application for commercial distribution. Thus, it represents an important intellectual property development for Sandia's Water Initiative.

## Accomplishments

We focused on the development of generic process modules necessary for quantifying the supply, demand, and allocation of water and other natural resources. Module formulation was loosely formulated according to two organizing themes: (1) water banking/leasing and (2) trade-offs between species preservation, urban development, and agricultural irrigation.

The surface and groundwater hydrology systems provide the backbone to the entire water resource system. We developed related modules and linked quantifying groundwater storage and flow, river routing, surface water-groundwater interaction (with and without pumping), and reservoir storage/operation. We coupled these hydrology modules to explicitly represent the movement and storage of water in the Middle Rio Grande Basin (MRGB). Specifically, we developed a model on a monthly time step and calibrated it for the entire Rio Grande and Rio Chama in New Mexico. The river system is discretized into 18 interacting reaches, including operations of the basin's seven reservoirs.

As part of the land surface model, modules for partitioning infiltration and shallow groundwater underflow were developed according to a three layer soil water bucket model. Additionally, we developed and integrated radiation balance models for computing incoming solar radiation with evaporation and transpiration models based on the modified Penman-Monteith equation and Hargreaves equations.

Aquatic species are key components in watershed management. Important dynamics associated with aquatic species vitality include population dynamics (births, mortality, migration) and impacts of environmental conditions (water availability and quality, pollution, dams, and diversions). We developed and tested a generic species module including these dynamics.

To quantify impacts of price on water demand, we developed agricultural modules relating water availability to crop yield and crop yield to economic return, and incorporated them into the MRGB model. Similarly, we developed urban and riparian valuation modules to understand their impact on water supply.

## Refereed

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*American Water Resources Association.*

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K. Cockerill, V. Tidwell, and L. Malczynski, "A Dynamic 'Toolbox' for Modeling Physical and Social Systems Related to Water Flows," presented at the International Society for Industrial Ecology Meeting, Stockholm, Sweden, June 2005.

V. Tidwell, D. Brookshire, A. Demint, D. Coursey, K. Carpenter, and J. Roach, "Towards a Market Based System for Water Resource Allocation," presented at the Fall Meeting of the American Geophysical Union, San Francisco, CA, December 2004.

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J. Roach, V. Tidwell, and K. Lansey, "Course Resolution Modeling of the Upper Rio Grande for Alternative Futures Scenario Evaluation," presented at the Fall Meeting of the American Geophysical Union, San Francisco, CA, December 2004.

V. Tidwell and H. Passell, "Decision Tools for Water Planning," presented at the New Mexico Rural Water Association Annual Meeting, Albuquerque, NM, April 2005.

# Predicting System Performance of Proton-Exchange Membrane Fuel Cells: Computational Modeling with Experimental Discovery and Validation

*67053*

*K. S. Chen, D. Ingersoll, C. J. Cornelius, D. R. Noble, N. P. Siegel, M. A. Hickner*

## Abstract

Hydrogen fed proton exchange membrane fuel cells (PEMFCs) are rapidly emerging as a viable technology for generating clean alternative power for automotive and stationary applications. However, several key technical challenges must be addressed before PEMFCs can be employed to routinely power our automobiles and homes. First, liquid water, produced by cathodic oxygen reduction reaction, must be carefully managed in order to obtain optimal PEMFC performance at practically high current loads. Sufficient water is needed in the membrane to maintain its conductivity, whereas too much liquid water can result in "flooding" in the gas diffusion layer (GDL). Flooding prevents oxygen from reaching reaction sites and causes performance deterioration. Second, effects of non-uniform hydrophilic and hydrophobic pore surfaces in cathode GDL and effects of hydrophilic cathode flow channel surfaces on PEMFC performance must be quantified. Third, the membrane electrode assembly (MEA) degradation and failure mechanisms must be elucidated. Though various piecemeal numerical treatments for PEMFCs have been reported in the literature, models are lacking that incorporate the essential phenomena to simulate PEMFC performance at practical operating conditions.

Our goal is to develop first-principle-based or phenomenological models to tackle these outstanding research challenges. Experiments will be carried out for phenomena discovery and model validation. Our objective is to develop an integrated PEMFC performance model by taking advantage of infrastructure in GOMA (a Sandia developed finite element computer code). Specifically, we propose to formulate a constitutive model for proton conductivity and validate it using experimental data. Phenomenological models will be developed to simulate the following: (1) transports of hydrogen/air in flow channels and GDLs, proton/water in membrane, and liquid water in cathode GDL; (2) hydrogen-oxidation/oxygen-reduction reactions in catalyst layers; (3) liquid water droplet formation, growth, and removal at the cathode-GDL/flow-channel interface; and (4) MEA degradation. Visualization experiments and performance measurements (current distribution and polarization curves) will be performed to validate the models.

## Accomplishments

We focused our efforts on elucidating the key phenomena of liquid water distribution, transport, and removal in a PEMFC. We also focused on developing and validating a baseline PEMFC performance computer model based on GOMA and publishing research findings.

More specifically, we accomplished the following: (1) we successfully carried out visualization experiments, using a specially designed experimental apparatus, to observe the formation, growth, and removal of the water droplets at the gas diffusion layer (GDL)/gas flow channel (GFC) interface of a simulated polymer electrolyte fuel cell cathode; (2) we refined and validated the simplified models, developed previously in FY 2004, for predicting the onset of liquid water droplet instability at the GDL/GFC interface; (3) we successfully carried out neutron imaging experiments at the National Institute of Standards and Technology's Neutron Facility to probe water distribution within an operating PEMFC; (4) we completed the development of a baseline PEMFC performance model based on GOMA; (5) we performed current-distribution and polarization-curve measurements using a specially designed and fabricated segmented PEMFC cell; (6) we carried out limited model validation using performance data obtained in our laboratory; (7) we investigated the effects of a microporous layer on liquid water transport in a PEMFC and documented the research findings in a refereed publication; and (8) we developed a non-isothermal multi-dimensional PEMFC model and used it to study the effects of anisotropic GDL thermal conductivity on temperature distribution.

## Refereed

K.S. Chen, M.A. Hickner, and D.R. Noble, "Simplified Models for Predicting the Onset of Liquid Water Droplet Instability at the Gas Diffusion Layer/Gas Flow Channel Interface," *International Journal of Energy*

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M.A. Hickner and K.S. Chen, "Liquid Water Droplet Instability Under Shear Flow on Porous Gas Diffusion Media for Fuel Cells," presented at The 2nd Asilomar Conference on Advanced Materials for PEM Fuel Cells, Asilomar, CA, February 2005.

K.S. Chen, M.A. Hickner, and D.R. Noble, "Toward Elucidating Key Phenomena That Control PEM Fuel Cell Performance: Computational Modeling with Experimental Discovery and Validation," presented at the Sixth Biennial Tri-Laboratory Engineering Conference, Monterey, CA, September 2005.

K.S. Chen, "Elucidating PEM Fuel Cell Performance via Computational Modeling and Experimental Investigation," presented at the New Mexico HyTeP Hydrogen & Fuel Cell Technology Workshop, Santa Fe, NM, September 2005.

# Silicon Field Emission Electric Propulsion (FEEP) Arrays Powered by Orbital Nuclear Reactors

*67055*
*R. X. Lenard*

## Abstract

The field emission thruster is a very high efficiency, space-proven device constructed using conventional fabrication techniques, where up to three emitter/extractor pairs are involved. The indium-Field Emission Thruster (In-FET), when constructed employing microelectromechanical system (MEMS) techniques, should provide very high power density in addition to high efficiency. However, each emitter/extractor pair is limited to ~10 microamps of current, requiring millions of emitter/extractor pairs for high power spacecraft. The potential significant payoff of this thruster was recognized by the Marshall Space Flight Center, which committed to testing a field emission electric propulsion (FEEP) array from Sandia.

Technically, the project achieved substantial advances:

(1) Formed an integral 4 material emitter base.

(2) Etched and machined the extractor.

(3) Coated the extractor with tungsten.

(4) Detailed, etched, and machined the tank segment.

(5) Coated the tank segment.

(6) Etched down the emitter substrate, exposing emitter chimneys to the correct height.

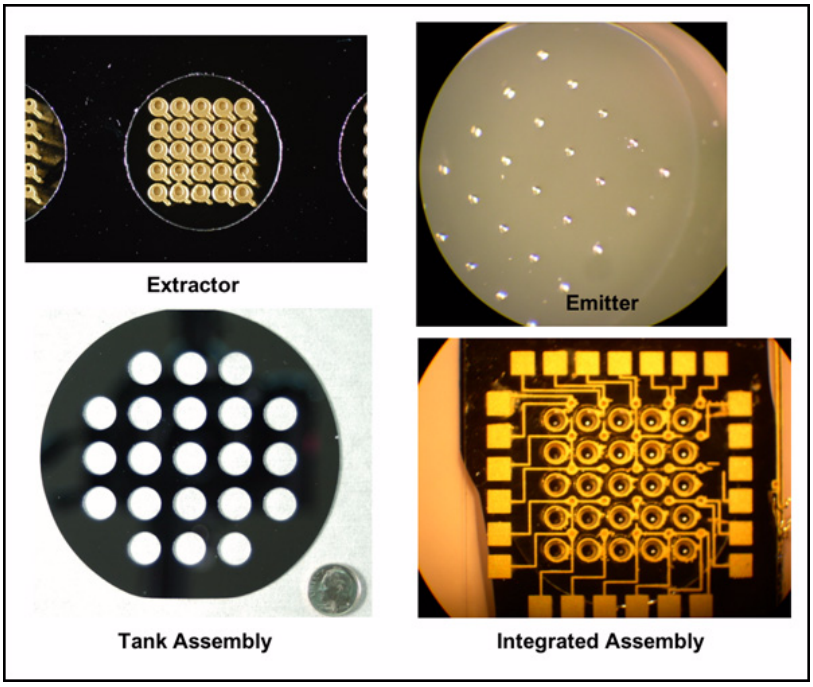
(7) Found methods to wet sputtered tungsten with indium.

(8) Built up a complete basic In-FET assembly to validate assembly processes.

(9) Found a way to electroplate tungsten, even on silicon (it unfortunately forms W-Si), but we have workarounds in progress.

(10) Successfully designed and fabricated a complete MEMS test assembly to physically match the test equipment of our CRADA partner, the Austrian Research Centers in Seibersdorf (ARCS).

Our present technical challenge is to get indium to wet the tungsten. Once that is done, our team will be ready to wet the emitters and capillaries. Subsequently, we will four-step bond the emitter, extractor, spacer, tanks, and backplate at the wafer level, dice the unit, and send it to ARCS for testing.




*Test bonding of integrated assembly.*

## Accomplishments

Accomplishments included:

(1) Developing preliminary design for In-FET.

(2) Developing preliminary design for test mounting apparatus.

(3) Designing tungsten cleaning glove box and laboratory.

(4) Completing the safety, pressure, electrical, and environmental documentation for the wetting laboratory.

(5) Completing detailed design of emitter, extractor, tank, backplate, and riser for the In-FET.

(6) Designing mask works for the emitter, extractor, tank, riser, and tank lid.

(7) Bosch-etching and back-filling emitter holes with $SiO_2$, polysilicon, and SiN with a final coat of tungsten.

(8) Demonstrating wetting of sputtered tungsten surfaces with indium in a wetting laboratory.

(9) Identifying a method to electroplate silicon with tungsten. Electroplated 25 sample pieces.

(10) Bonding tank and emitter test articles.

(11) Etching emitter chimney structure with two-stage, hard-soft photo-etch materials.

(12) Identifying a plasma cleaning process to clean tungsten metal surfaces.

(13) Developing getter gas process to clean fluorine out of chemical vapor deposited tungsten surfaces.

(14) Completing preliminary design for test jig interface equipment.

(15) Completing detailed design of cover plate, selector rings, contactor plates, and contactor assembly for interfacing with ARCS's test apparatus.

(16) Fabricating electrical connection device to energize individual emitter/extractor pairs of the In-FET test device.

(17) Performing test bonding of a complete In-FET thruster assembly, including extractor, emitter, tank, riser, and backplate.

(18) Performing anodic bonding of six pyrex/silicon substrates into a single integrated structure. The success of this approach is in the verification stage. This practice has never been accomplished before. If the advance is verified, our team will submit a patent application for this important process.

(19) Achieving some wetting success with sputter cleaned tungsten.

(20) Metal Oxide Chemical Vapor Deposition (MOCVD) trimethyl indium wetting experiments are still not conclusive.

(21) Testing 5x5 array at ARCS.

(22) Analyzing and comparing results with predictions.

(23) Designing 10x10 array incorporating all lessons learned.

## Refereed

R.X. Lenard, S. Kravitz, and M. Tajmar, "Recent Progress in Silicon-Based MEMS Field Emission Thrusters," presented and in *Proceedings of the Space Technology Applications International Forum*, Albuquerque, NM, February 2005, vol. 746, pp. 954–964.

# Advanced Fuel Cell Reactor for the Direct Cogeneration of Electricity During Selective Partial Oxidation of Hydrocarbons

*67056*

*A. H. McDaniel, J. E. Miller, R. E. Loehman, S. F. Rice*

## Abstract

A fuel cell operates on the principle that either hydrogen or hydrocarbon fuels be completely oxidized to water and carbon dioxide while generating electricity. In addition to producing electricity, an advanced fuel cell reactor would also control oxidation to produce hydrogen or other valuable oxygenated hydrocarbons. The proposed electrocatalytic membrane reactor will make novel use of fossil fuels directly in solid oxide fuel cells (SOFCs). At the same time, it would address the broader needs of energy security by replacing more conventional, less efficient fuel reformer and selective oxidation processes. A research team, which combines expertise in materials and advanced diagnostics, is focused on realizing these objectives. Activities involve the synthesis and evaluation of novel membrane electrode assemblies activated for selective oxidation of hydrocarbons. The project involves challenging materials development, catalyst formulation, processing issues, advanced diagnostics, and experimentation. The main objective is to prototype the reactor concept for a select class of partial oxidation reactions to provide Sandia with a unique position in an area that has not been effectively exploited for SOFC applications. Unlike existing Department of Energy (DOE) programs targeting auxiliary power, the SOFC-like concept for combined chemical production and electrical generation is an emerging field.

## Accomplishments

Our research team characterized both the non-electrochemical and electrochemical behaviors of novel anode catalyst formulations. Further, the team investigated the application of surface Raman spectroscopy to probing anode surfaces in situ, synthesized and evaluated various catalysts formulations for partial oxidation of methane (POM), and developed and refined methods for in-house preparation of membrane electrode assemblies (MEAs). The team made pertinent observations on POM that lend credibility to the initial hypothesis that the extent of hydrocarbon oxidation may be controlled using electrochemical means.

We evaluated prototyped anode catalyst formulations and MEAs in both novel and conventional reactor geometries using laser based and impedance diagnostics, traditional potentiometric and amperometric methods, and mass spectrometry. We used a hot-wall air breathing configuration predominantly to characterize both anode-only thick films and complete MEAs. The reactor consists of either an MEA or an alumina-supported anode film atop a ceramic substrate, which is glass sealed to the end of a ceramic tube. Gas mixtures are fed into the closed end of the tube. External heating is achieved by placing the tube assembly into a furnace. Ambient air is used as oxidizer with the MEA running as a true fuel cell. The cold end of the tube has an optical port and sufficient view of the surface to perform Raman scattering measurements. A commercial computer controlled potentiostat (Gamry Instruments PCI4/750) is used to execute all electrochemical measurements.

The motivation for developing the pulsed Raman diagnostic was to explore the detailed nature of the oxidation state and structure of the yttria-stabilized zirconia (YSZ) surface in situ, at temperature, while partial oxidation processes were active. In particular, the motivation was to explore whether the chemical reactivity of the mobile oxygen, transported through the membrane, is distinct from other oxygen species in the system (bulk lattice oxygen or atomic oxygen resulting from chemisorption). We obtained simple ambient temperature spectra of the YSZ surface. We also obtained a variety of candidate anode materials in two different oxidation states, post calcined in air and after high temperature reduction in hydrogen. The Raman spectrum of YSZ (zirconia with approximately 10 atom% yttrium) consists of all six Raman allowed resonances of tetragonal YSZ. It is very well defined and can be easily measured under reacting conditions. As for the Ni:YSZ material, the Raman spectrum is much weaker than that of clean YSZ, which is disappointing but not entirely surprising. The probative value of surface Raman applied to this system is yet unclear.

This year the team also gained proficiency in formulating anode powders in complete MEA fabrication.

We prepared a baseline Ni:YSZ composition, along with a family of doped derivatives composed of alkali metals, alkaline earth metals, transition metals, and lanthanides. We fabricated anode-only thick films and MEAs by powder processing, slurry formation, patterning of cathode-only half-stacks, optimizing multiple firing steps, applying patterned current collectors with attached metal wire conductors, and high temperature sintering and sealing of ceramic components.

Finally, we made pertinent observations on POM, which lend credibility to the idea that the extent of hydrocarbon oxidation may be controlled using electrochemical means. We observed an apparent difference in reactivity between chemisorbed oxygen and ion-pumped oxygen over an active MEA in the hot-wall reactor. $O_2$ was co-fed with $CH_4$ in a 4:1 fuel rich mixture to an anode operating at 900 K (fairly low temperature for SOFC). At open circuit potential (EOC), thermal POM occurs, producing $H_2$ and CO (in addition to small amounts of $CO_2$ and $H_2O$). When the cell bias was made more anodic relative to EOC, a pronounced increase in $H_2$ and CO production ensued with little discernible increase in $CO_2$. This result suggests that ion-pumped oxygen species are preferentially inclined to partial oxidative pathways when reacting with adsorbed hydrocarbon, which lends credence to our initial hypothesis.

# New Hydrogen Storage Material: Metal-N-H System

*67057*

*W. Luo, E. H. Majzoub, K. J. Gross*

## Abstract

Hydrogen storage is one of the most challenging issues for the hydrogen economy. In FY 2004, we successfully developed a new material: a Mg modified Li amide/hydride, which can deliver hydrogen at a pressure of 30 bar at 200 °C with hydrogen capacity of 5% by weight. This year, the goal was to search for catalysts to improve sorption kinetics. Several additives were tested, and no significant catalytic effect was observed. However, we found that the absorption rate depends strongly on the applied hydrogen pressure and that absorption rate can be improved significantly by increasing hydrogen pressure. We also investigated the reaction mechanism using x-ray diffraction (XRD) and Fourier transform infrared (FTIR) techniques. We found that the host elements (Li, Mg, or N) have to migrate in solid storage material in order to complete the hydrogen absorbing/desorbing process. This is different from traditional interstitial metal hydrides. This reaction mechanism may provide clues for future catalyst research.

## Accomplishments

In this project, we successfully developed a new hydrogen storage material by partial substitution of Li by Mg in the $LiNH_2$-LiH storage material proposed by Chen [1].

Major accomplishments are:

(1) We developed a viable hydrogen storage system capable of:

(a) Delivering hydrogen of 30 bar at 220 °C with hydrogen 5.2% by weight and

(b) Absorbing hydrogen reversibly and has been cycled at 220 °C with capacity loss only 11%.

(2) We also investigated the reaction mechanism by using XRD and FTIR techniques. For the $LiNH_2$-LiH storage system, it was understood that the desorbed material was $Li_2NH$, as shown in the following equation: $LiNH_2 + LiH \Rightarrow Li_2NH + H_2$.

For the Mg modified storage system, however, the desorbed species had never been identified. In this study, we found that the desorbed material was not lithium imide ($Li_2NH$), magnesium imide (MgNH), or a mixture of both. Rather, it was a binary imide, $MgLi_2(NH_2)$. After a full cycle of sorption, the reabsorbed material was a mixture of $Mg(NH_2)_2$ and LiH, and the true reversible reaction is between ($MgLi_2(NH)_2 + 2H_2$) and ($Mg(NH_2)_2$+2LiH), as shown in the following reaction: $2LiNH_2 + MgH_2 \Rightarrow MgLi_2(NH)_2 + 2H_2 \Rightarrow Mg(NH_2)_2 + 2LiH$.

(3) We also identified that this is solid phase reaction, so the reaction rates strongly depend on the particle size of the reactant. Since there are two reactants in the hydrogenated material, the mixing of these two species is very important in achieving higher capacity and higher sorption rates.

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## Refereed

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# Intrusion Detection for Wireless Networks

*67058*

*J. Van Randwyk, R. P. Custer, E. J. Lee, D. Kilman, E. D. Thomas*

## Abstract

We developed an intrusion detection and monitoring architecture for wireless networks. The architecture specifically applies to Institute of Electrical & Electronics Engineers (IEEE) 802.11b/g standard wireless networks, the type of networks referred to as "Wi-Fi."

Our research was broken into several distinct areas that all contribute to a complete architecture. These include a study of radio frequency (RF) jamming and jamming mitigations, location-based authentication of network devices, three-dimensional (3-D) visualization of wireless networks, wireless network monitoring, and MAC (media access control) layer and RF-layer device fingerprinting.

Wireless networks are rapidly being deployed with attention often paid to the integrity and confidentiality of the data being transmitted, but without proper intrusion detection or monitoring on those same networks. We anticipate this research will give Sandia and other national labs within the Department of Energy (DOE) confidence to deploy wireless networks when using our architecture as a guide. This work could potentially generate a "best practices" guide to monitoring infrastructure wireless networks in the Department of Homeland Security (DHS), other federal agencies, and even private organizations.

## Accomplishments

We broke our research into several distinct areas that all contribute to a complete architecture:

We studied the RF layer of the 802.11b protocol, looking for ways to detect and mitigate attacks at that layer. We simulated RF-layer attacks and modeled the disruption of the wireless network. We showed that intelligent, directed jamming attacks are hard to mitigate.

We also studied methods for physically locating devices in a wireless network. We addressed location determination techniques, a model of location-based authentication, strategies and policies for dealing with location estimation system error, a realistic deployment of the system, attacks against the system, and possible defenses against these attacks. Our research showed that inexpensive and high-integrity location-based authentication is a practical solution to authentication issues.

Additionally, we studied new ways to visualize wireless networks. We developed tools and systems to capture wireless network traffic and display it in a 3-D environment. Looking at the networks in this new format allows security analysts to see previously unseen anomalies and, potentially, attacks. As far as we have been able to determine, the code written for these tools is the first of its kind. We are discussing the possibilities of making the code freely available via open source licensing.

We built a monitoring system for recording wireless network traffic and formatting it in a human readable form. This type of system is necessary for forensic analysis after a network break-in has occurred. The data recorded by this system allow a security analyst to recreate the state of a network at the time that the attack and/or compromise occurred.

We also explored the area of fingerprinting wireless devices, specifically wireless network interface cards. We studied fingerprinting devices at both the MAC layer and the RF layer. Our research into RF fingerprinting showed that this type of fingerprinting may be possible, an area not previously studied. Our work on fingerprinting devices at the MAC layer resulted in the development of a method for fingerprinting by analyzing the time in between probe requests frames. We found our observed data to be a function of the software driver being used for the given wireless interface card.

## Other Communications

R.P Custer, E.J. Lee, and J.A. Van Randwyk, "Using 3-D Network Visualization to Enhance Rapid Threat Recognition and Response," presented and in *Proceedings of Working Together: R&D Partnerships in Homeland Security*, Boston, MA, April 2005, CD-ROM.

R.P. Custer, E.J. Lee, and J.A. Van Randwyk, "Using 3-D Visualization to Enhance Understanding of Computer Network State," presented at the ACM SIGGRAPH 2005, the 32nd International Conference on Computer Graphics and Interactive Techniques, Los Angeles, CA, July 2005.

# Membranes for $H_2$ Generation from Nuclear Powered Thermochemical Cycles

*67059*
*T. M. Nenoff, T. J. Garino, A. Ambrosini, F. Gelbard*

## Abstract

Environmentally sound methods for the production of hydrogen are of great importance to the US as we progress toward the $H_2$ Economy. 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. Current studies aim at using high temperature systems (i.e., nuclear, solar) to drive thermochemical cycles for $H_2$ production. A promising candidate is the Sulfur-Iodine (S-I) cycle in which the decomposition of $H_2SO_4$ into $O_2$, $SO_2$, and $H_2O$ at 850 °C is the first step. Removal of $O_2$ from this reaction would push the equilibrium towards dissociation, thus increasing the overall efficiency. The demanding conditions of high temperature and the oxidizing environment of the decomposed $H_2SO_4$ require a robust separations membrane.

Perovskites are potential materials for oxygen separation membranes, owing to their high temperature stability, the ability to stabilize oxygen nonstoichiometry, 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 focused on dense ceramic membranes based on the double-substituted perovskites, $AA'BB'O_{3-d}$. Materials in the family $La_xSr_{1-x}Co_yB'_{1-y}O_{3-d}$ (where B' = Fe or Mn) show promise for such separations. We synthesized and characterized several perovskites in each family and are in the process of testing the oxygen permeability of membranes fabricated from these materials.

## Accomplishments

To date, the project consisted of four major research areas: (1) literature surveys and thermochemical team discussions; (2) materials synthesis and characterization of ceramic oxide phases for high temperature $O_2$ separations; (3) conductivity apparatus design, construction, and testing; and (4) benchscale permeation unit design and construction.

We chose perovskites as potential materials for oxygen separation membranes owing to their robustness, ability to stabilize oxygen nonstoichiometry, 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.

- Synthesis and Characterization:

We structurally characterized and tested synthesized materials for phase purity by powder x-ray diffraction (XRD). We performed elemental analysis via inductively-coupled plasma spectroscopy on several of the samples to assure that their cation stoichiometries coincided with expected values. For absorption/desorption studies, we ran multiple-cycle thermogravimetric analysis (TGA). First, we cycled the temperature between 50–850 °C under an $O_2$ atmosphere. We followed this by cycling between $O_2$ (high $pO_2$) and Ar or He (low $pO_2$) atmospheres at 850 °C. We ran XRD studies before and after thermal study samples to confirm that the material retained its original perovskite structure. To date, our most promising samples come from the perovskite family, $La_{0.1}Sr_{0.9}Co_{1-x}Mn_xO_{3-d}$ (LSCM), where 0.1 = x = 0.3, and we concentrated much of our attention on these materials. Oxygen stoichiometry in several LSCM samples determined by iodometric titration resulted in values of 0.20 = d = 0.24.

We investigated the composition and adsorption and desorption properties of single-phase compounds we synthesized. We demonstrated that such TGA analysis is an effective screening method for potential oxygen permeability. Materials with high average weight loss during temperature cycling and variation of the partial pressure of oxygen are the materials of greatest interest for development into membranes. Samples showing the highest weight losses as a function of tem-

perature and partial pressure of oxygen include those in the LSCM family and similar double-substituted perovskites (YSCF, LSCF, LSCCr). In addition, we measured yttria-stabilized zirconia (YSZ) as a "baseline."

- Conductivity:

Four-probe conductivity measurements on the various oxide phases versus temperature and $pO_2$ were performed on LSCM and LSCF materials. The overall conductivity of the materials is in the range of 10–102 S/cm, more than three orders of magnitude greater than that of YSZ. Such a high conductivity implies a high degree of electronic (as opposed to ionic) conductivity. Ionic conductivity is probably also present, based on the literature and sorption properties of the material, but it is overshadowed by the electronic character.

- Permeation:

We fabricated self-supported 0.5" diameter ceramic membranes by uniaxially pressing LSCM powders and sintering them at 1250 ºC. We completed the construction of the permeation unit. We built the unit out of Inconel 600 alloy, which is stable at the high temperatures necessary for our permeation experiments. We plumed a micro gas chromatograph to analyze the output stream. Leak and sealant tests are currently in progress.

## Refereed

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A. Ambrosini, T. Garino, A. Navrotsky, 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," presented and in *Proceedings of the Solid State Ionics 15th International Conference*, Baden-Baden, Germany, July 2005, CD-ROM.

# Microfabricated BTU Monitoring Device for System-Wide Natural Gas Monitoring

*67060*

*A. Robinson, M. W. Moorman, R. P. Manginell, R. T. Miner, J. A. Ellison*

## Abstract

The natural gas industry is seeking automation of pipeline processes and distribution. The development of an inexpensive monitoring device for the gas heating value, the British thermal unit (BTU), is desirable for pipeline system analysis and gas accounting. As natural gas is sold by the BTU, improved analysis will produce more equitable trade between buyers and sellers by reducing unaccounted for (UAF) gas content. A 10% improvement in UAF gas accounting through improved sampling and analysis will benefit the gas transport industry by approximately $45 million annually. In particular, industrial users will benefit through continuous feedback of physical gas properties during use with gas turbine engines used for power and electricity generation.

For market acceptance, a monitoring system must have low cost. According to a market analysis, widespread commercial implementation will only occur below a threshold cost of approximately $5000 per instrument. We can achieve this unit cost by using microfabricated analytical components. We assembled a prototype BTU monitoring instrument using existing and modified microfabricated components. In conjunction with a commercial injection valve, the instrument uses a silicon microfabricated, packed gas chromatography (GC) column. A microfabricated pellistor serves as the detector. The micro-pellistor is used to sense combustion at its catalytic surface through monitoring the power required to keep the device at a constant elevated temperature. A second element serves as a reference. Batch microfabrication techniques will allow these components to be manufactured in production quantities at a low cost.

This combination allows calorimetric measurements to be made with a 1 minute run time and a 1.5 minute cycle time. It is hoped that an instrument of this type will replace month-long sample tank collections at remote natural gas production wells and provide near real time data at pipeline switching and metering stations and at industrial sites.

## Accomplishments

This project encompassed three major tasks in concurrent development. The first task tailored a microfabricated GC column to separate injections of natural gas. The second task experimented with micro-hotplate pellistors to respond to natural gas compounds, with emphasis placed on rapid response to chromatographic exposures. Third, we fabricated a complete field-portable prototype gas chromatographic instrument using these and other commercially available components.

We performed natural gas separations using Sandia's packable chromatography columns microfabricated from silicon and glass. We packed the columns with various chromatographic materials commonly used in natural gas separations. We then determined the performance of the columns, their separation ability, and the analysis time under various temperatures and pressures. We found HayeSep polymers (Supelco, Inc.) to produce the best performance. A 19 cm column with HayeSep B completely separated all of the compounds by carbon number and nearly separated the isomers. Analysis time was two minutes. A 15 cm column with HayeSep D can separate all of the compounds by carbon number, with little isomer separation, in less than one minute. Thermal ramping of the low mass silicon column speeds the run time considerably, while maintaining excellent resolution. Partial separation of the isomers produces a maximum 0.009% error in the BTU (heating) value for each one percent of C4+ hydrocarbon in the mixtures. The chromatography packing showed no degradation after 500 analyses with air carrier and thermal cycling from 80 to 120 $^{o}$C. It also showed excellent reproducibility of peak position from run to run.

We made efforts to understand the mechanisms of catalytic combustion for the micro-pellistor sensor and to optimize combustion behavior during chromatography. This involved optimizing combustion efficiency by optimizing flow rate, dead volume of the sensor, and sensor temperature. Sensor temperature is inversely related to lifetime of the supporting membrane. We determined that an average surface temperature of 480 °C maximizes combustion while providing life-

times of weeks or longer. Dead volume did not seem to affect the combustion percent, though this does cause spreading of chromatography peaks. After flow rate optimization in the range of 30 to 40 mL/min, we determined that combustion efficiency does not exceed 35% at this time, though the combustion percent for each compound is highly reproducible under a given set of experimental conditions. Future work will address the issue of incomplete combustion, which will increase the sensitivity of the sensor. We also performed some work on sulfur poisoning of the catalyst material. While we found carbon disulfide to poison the catalyst, we can reverse the effect through continuous exposure to methane at combustion level temperatures.

We combined the micro-packed chromatography column and the pellistor sensor with a commercially available pneumatic injection valve in a prototype field portable GC instrument. A custom electronics board provided independent PID (proportional-integral-derivative) temperature control of the GC column and pellistor fixture. A second custom board provided thermal control of the pellistor and reference sensors with outputs proportional to hotplate power. We controlled and read these two boards through a commercially available data acquisition board. We controlled the board and all instrument functions through custom LabVIEW programming. We fixed these components inside a travel suitcase, which also holds a small laptop. The only external component is a small tank containing two cubic feet of zero air, enough for 100 hours of operation. As this instrument reached final assembly at the end of the project, we have yet to test it in its entirety.

## Refereed

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## Other Communications

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# "Tunable" Ion Conductors for Low Temperature Oxide Based Fuel Cells

*67061*

*T. M. Nenoff, T. J. Garino, J. D. Pless*

## Abstract

We propose a new method for the synthesis of perovskite materials with high ion conductivity for various applications in low temperature ceramic based fuel cells for use in the US $H_2$ economy. Oxide-based fuel cells operate at high temperatures with advantages that include high efficiency, flexible fuel types, and inexpensive non-noble catalysts. Conversely, high temperatures lead to component breakdown and large size heating units. A major materials breakthrough is needed in this technology to lower the operating temperatures, while retaining high catalytic and diffusion activity, to use a variety of fuel sources and produce high surface area phases.

In this project, synthesis and characterization of tunable perovskite ceramics is under way and directed toward the control of strength and temperature of dielectric constants with ionic conductivity. Traditional methods of synthesis involve high temperature oxide mixing and backing. We are focusing on a new method of synthesis involving: (1) low temperature hydrothermal synthesis of metastable porous phases with tuned stoichiometry and element types, and (2) low temperature heat treatment to build exact stoichiometry perovskites with the desired vacancy concentrations. This flexible pathway can lead to compositions and structures not attainable by conventional methods. These materials are being studied by high temperature ionic conductivity measurements to better delineate stability and stoichiometry/bulk conductivity relationships. This in turn will lead to a predictive model for the possible extent of substitutions.

## Accomplishments

The project consists of four major research areas: (1) Sandia octahedral molecular sieves (SOMS) and novel SOMS synthesis and characterization, (2) SOMS-derived perovskite synthesis and characterization, (3) conductivity testing of the sintered phases, and (4) calorimetry of the phases. Results from the SOMS and perovskite syntheses include compositions now made from either alkoxides or oxides (allowing for future commercial scale-up). Furthermore, we extended the phase space to include Na/Nb, Ta/Ti, Hf, Mo(V), Zr, Ge, and Te. In addition, we ion exchanged cations of varying oxidation states (i.e., $Li^+$, $Co^{2+}$, $Fe^{3+}$) into SOMS, giving new compositions. We characterized the samples using powder x-ray diffraction, electron microscopy, thermal and elemental analyses, conductivity measurements, and calorimetry. The chemistry, conductivity, and calorimetry allow us to understand the structure/property relationship of the resultant ion-conducting materials. Furthermore, in anticipation of FY 2006 milestones, we began discussions about potential application.

We standardized the synthesis of the SOMS phases to a hydrothermal procedure: (1) $Nb(OEt)_5$ and $M(OR)_4$ alkoxides are mixed and sealed in a glove box; (2) separately, NaOH is dissolved in $H_2O$ and placed in a 23 ml Parr Teflon liner; (3) the metal alkoxide mixture is added to the aqueous NaOH; (4) the liner is placed inside a Parr reactor and sealed; (5) the reactor is placed into an oven at 170 °C for four hours (the 20% titanium substituted SOMS reacts for 96 hours); and (6) the products are filtered and washed with deionized water. Perovksite phases are synthesized either by direct low-temperature hydrothermal methods or heat-treatment of SOMS metastable phases.

We prepared samples of $Na_2Nb_2O_6$-$H_2O$ and 20% Ti-SOMS for in situ inelastic neutron scattering (INS) experiments in the temperature range of 20 to 800 °K using the filter difference spectrometer (FDS). We washed the samples with $D_2O$ in an effort to exchange the protons for deuterium atoms needed for neutron studies. This allowed for the assignment of the vibrational modes (O-H stretching and bending) related to the H-bonds for water molecules and hydroxyl moieties and will elucidate the effects of Ti substitution on the ability for ion exchange.

We measured and compared the conductivities of hydrothermally synthesized perovksites and ion exchanged 20% Ti-SOMS-derived perovskites (sintered at 1200 °C, 1 hour) to the standard yttria-stabilized zirconia (YSZ). The data indicate that the conductivity in air increases as transition metals are substituted into the framework structure. The amount of metal substitution into the framework also affects the conductivity, indicating atomic scale variations effect-

ing bulk properties. Furthermore, the conductivity decreases in the samples that were ion exchanged into 20% Ti-SOMS (heat treated to perovskites), except for the $Mg^{2+}$ exchanged sample. The decrease in conductivity is unexpected as the exchange of monovalent $Na^{+}$ for divalent cations ($M^{2+}$) might have resulted in cation defect sites in the perovskites. We speculate that charge compensation of the perovskite might be occurring through the incorporation of oxygen atoms. If oxygen atoms are incorporated, it would probably lead to a decrease in the conductivity. Another reason might be distortions in the perovskite structure resulting from differences in the atomic radii.

We measured the heats of formation for the SOMS variations by high temperature oxide melt solution calorimetry. Samples with more positive $\Delta H_{f,ox}$ values are more stable and contain fewer defect sites. The calorimetric data correlate with the conductivity data, thus indicating that the conductivity increases with an increasing amount of defects. Furthermore, we found that the thermodynamic data of the alkali earth metals correlates with the cationic radii. Characterization of the materials is ongoing and will aid in further synthetic directions.

## Refereed

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# Nuclear Nanobatteries: On-Board Power for Independent MEMS Devices

*67062*

*J. A. Crowell, D. F. Cowgill, D. A. Chinn, F. P. Doty, R. A. Causey, C. Munoz, C. M. Cuppoletti*

## Abstract

Size reductions of microelectromechanical systems (MEMS) have not been matched by similar reductions of their power supplies, limiting the potential for independent microsystems. Radionuclides, which have energy densities three to five orders of magnitude greater than chemical batteries, may offer a solution that can be integrated into these devices.

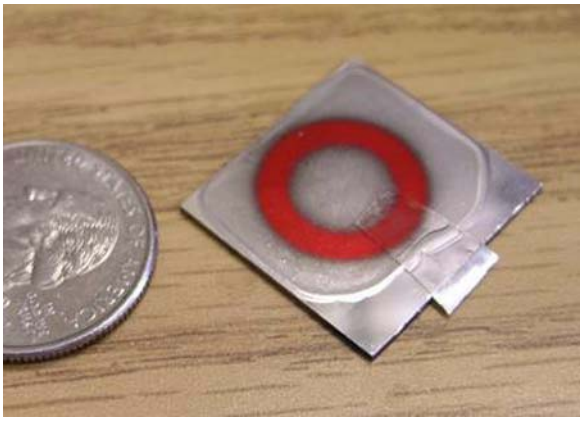
The goal of this project was to demonstrate a microscale, radionuclide powered battery using organic semiconductor junctions, specifically heterojunctions of pi-conjugated materials (PCMs). These materials offer good radiation hardness, ease of fabrication, flexible structure, and a wide choice of materials and properties. While succeeding in significant advances in materials development and junction design, poor conversion efficiency, perhaps arising from charge diffusion in the materials, prevented demonstration of power levels sufficient to drive MEMS devices.

In the section below, we summarize our developments in polymer junction design and the remaining challenges to a successful polymer based radionuclide battery. Our success in producing so-called bulk heterojunctions was greatly assisted by our development of a novel solvent system for filtering troublesome particulate impurities.

## Accomplishments

A significant portion of the work on this project involved characterization of polymer films. The performance of these materials is extremely sensitive to the processing conditions, so we gave significant attention to developing processing methods that produced films with optimal and consistent characteristics.

Two developments produced heterojunctions with favorable electrical characteristics. One of these developments was the production of “bulk” heterojunctions. These consist of a single layer of blended PCMs with single-walled carbon nanotubes sandwiched between dissimilar metal electrodes. These devices exhibited superior performance when compared to the bi-polymer heterojunctions produced initially.



*Test device. The red ring is bare polymer exposed when the O-ring mask was removed. The active area is inside this ring.*

The other development was the discovery of a novel solvent system that allowed us to filter troublesome particulate impurities from our heterojunction materials before spin casting. This discovery resolves a long standing problem plaguing the fabrication of polymer electronics. We obtained these characteristics using a solvent system (patent applied for) that allows for much more concentrated solutions (allowing for thicker films) and dissolves both the conjugated polymers and nanoparticles (our two charge transport materials).

The attachment of electrical leads initially presented difficulties. Placement by hand of the wire lead developed concentrated pressure against the thin layered structure, depressing the surface electrode down into the polymer layer until it shorted against the opposing electrode. An arrangement of tape as a cushioning layer over part of the device resolved this problem.

We investigated the effects of spin casting and drop casting against initial 4-point probe screening, as well as a variety of solvents and filtering techniques. We also looked at various compositions of PCMs and junction configurations.

The conclusions from the initial qualifying test devices were that bulk heterojunctions cast from styrene yield films with the most favorable properties. Our

next steps were testing with the electron-beam. We tested several devices, each having different metals or charge injection layers, to address issues that arose during e-beam testing. All the metals were sputtered in our Kurt J. Lesker sputtering tool. Bottom contacts were typically > 100 nm thick; top contacts were 10–50 nm thick. LiF was evaporated to an approximate thickness of 0.4 nm as a charge injection layer at the Al contact. PEDOT-PSS is a conductive polymer used as a charge injection layer at the high work function contact. If heat was indicated, we heated films on a hot plate at 200 °C to enhance ordering and/or phase segregation of the polymer layer. Top contacts were generally sputtered on the device through an O-ring mask to define the active region of the device. The active area was a ring of bare polymer exposed when we removed the O-ring mask.

We measured voltage across, and current through, a resistive load produced by exposure of the devices to an accelerated electron beam. The electron gun is a particularly useful tool since it allows us to characterize device response to nearly mono-energetic, mono-directional electrons. By taking measurements at numerous energies and incident angles, we can fully map the device response and predict the performance of the device with a beta energy spectrum and isotropic source.

Unfortunately, the conversion efficiencies of the devices proved to be below 0.002%, orders of magnitude below the targets we had set. As a result, we decided not to produce devices with a radionuclide source as we would expect them to produce power far below our target of 200 nW.

# Integrated Tunable Light Sources for Miniature Sensors

*67063*

*R. P. Bambha, S. E. Bisson*

## Abstract

Compact, rugged mid-infrared light sources will enable new instruments for widespread monitoring of natural gas delivery systems and leak detection by first responders at disaster sites. The devices will be fabricated in quasi-phase-matched (QPM) materials, which Sandia is exploiting in current sensors. Sandia has developed in-house capabilities to make QPM materials. The additional ability to design microscale waveguide structures, which can be fabricated in the QPM materials, is a major advance toward new devices for demanding applications. The main advantage of waveguides in frequency conversion results from the confinement of the interacting beams to a small area, which enhances the conversion efficiency. Furthermore, integrated photonic structures would allow additional functions, such as switching and routing multiple wavelengths on the device, to increase the capabilities and commercialization potential of compact sensors. The ability to build nonlinear waveguide devices could also be applied to visible light generation, which could enable new detection systems for homeland security applications.

## Accomplishments

During the course of this project, we determined that the ability to model the fabrication and behavior of the waveguide devices is the greatest barrier to the development of these devices. Fabrication services for waveguide devices are available commercially, but design services for difference frequency mixing devices are not commercially available. We focused our efforts on developing the ability to design and model the devices and to perform laboratory measurements on devices fabricated elsewhere. We compared our model results to laboratory measurements we made on devices fabricated at Stanford University.

Waveguides in $LiNbO_3$ are formed by diffusing protons through a mask on the surface of a polished $LiNbO_3$ wafer. Because the diffusion processes have complicated nonlinear, time dependent behavior and because waveguides have a complicated index of refraction profile, numerical modeling is a crucial aspect of the waveguide development process. The predominant numerical method we used was finite element analysis. The diffusion equations that we used are semi-empirical and were developed at Stanford University. They are based on an ion exchange diffusion model that results in a concentration-dependent diffusion coefficient, which makes the diffusion process non-linear. Time dependent numerical modeling is required to predict the diffusion profile. The software we use is a general purpose finite element analysis package known as Femlab. From the diffusion analysis, we develop a model of the actual waveguide profile that results from the fabrication conditions that include the mask opening width, diffusion temperature, and initial concentration.

We also use the finite element method for electromagnetic analysis of the waveguide to determine the eigen modes of a particular device. Using the modeled waveguide diffusion profile, we can calculate the propagation constants for the different waves. We then use the propagation constants to determine the poling period of the periodically poled lithium niobate crystal, which allows the interacting waves to be quasi-phase matched for maximum conversion. With the electromagnetic fields determined for all the interacting waves, we can calculate the expected conversion efficiency. This provides us with a means of predicting the performance of the device. Our model showed good agreement with experiments made on devices designed to mix 1.064 microns and 1.55 microns to produce outputs in the ~3 micron region. We also used the model to design devices in regions where the efficiency of bulk devices is typically too low to be practical. Using our model, we designed devices for generating infrared light at 4.3 microns, where $LiNbO_3$ has poor transparency using input lasers at 1.13 and 1.54 microns. This design is complicated by the substantial difference in the sizes of the modes for the input and output waves. We predict that the 4.31 micron radiation will be produced with an efficiency of $5500\%/(W^2m^2)$ without attenuation. Including attenuation, we predict that the device will be able to produce approximately 50 microwatts output for a pump power of 1 W and a signal power of 10 mW, which indicates that a waveguide is a viable option for a spectroscopic light source at 4.3

microns. We used another numerical technique, the beam propagation method (BPM), to model the taper section used to expand the single mode input to the mixing region. The BPM model is used to design tapered sections of the waveguide to transform the size of the mode with acceptable insertion loss and taper length.

# Energy Systems Analysis Framework and Modeling

*72716*

*G. W. Kuswa, O. H. Paananen, J. D. Brewer, M. M. Valdez, , J. V. Zuffranieri, J. Y. Tsao, T. E. Drennen, S. A. Jones*

## Abstract

The purpose of this project was to improve Sandia's multidisciplinary capabilities in energy systems analysis. The first year of the project, FY 2004, saw the development of a framework for comparison of attributes associated with various energy options. We examined hydrogen in some detail, particularly looking at the required infrastructure and the attendant environmental issues that would result. The initial product is documented in a series of unpublished white papers that span a broad range of topics, including the successes and failures of past modeling studies, sustainability, oil dependence, energy security, and nuclear power.

The second year of the project, FY 2005, saw the refinement and further development of the framework and focused on understanding how various electricity generating options can best serve US needs. The project provided inter-comparisons of present electric power sources. We developed a computer tool to compare various options based on cost and other attributes such as technological, environmental, social, and policy constraints. Some new features developed under this project include: (1) unique formalism for combining scores of dissimilar metrics, such as cost and environmental factors, into a single score that accounts for specific judgmental factors of the evaluating audience; (2) a detailed report describes how individuals arrive at judgments concerning major decisions and how they determine what risks they are willing to assume. The latter topic, summarized here, sets the stage for training practices that may help Sandia gain public acceptance for technologies.

Another topic of note is that studies during the project indicated the desirability of closing the carbon cycle for transportation fuels by using carbon dioxide and water as the feedstock to synthesize hydrocarbon fuels using nuclear or renewable energy sources. This would avoid the need for a costly new hydrogen infrastructure.

## Accomplishments

In FY 2005, we focused on comparing the relative merits of major electric generation methods based on the multi-criteria framework developed during the inception of the project. In this context, we developed a model in PowerSim that enables a group of people to engage in animated and informed discussion about the relative merits of electric generation alternatives. The model can engage the interest of expert and lay audiences alike. This model is of interest to several officials in the Department of Energy, and we believe that it will present an opportunity to secure support for expanded similar studies, such as alternative transportation energy sources.

We assembled participants representing a wide range of skills and viewpoints, such as renewable energy, fossil energy, national security, research, risk analysis, and nuclear energy. We also supported the involvement of an external energy economist/modeler and a visiting scientist from the German Aerospace Lab, and this helped the team to broaden its perspectives. The product of this effort was a series of white papers on many topics, including sustainability, the role of non-governmental organizations in influencing policy, how the public and officials reach decisions on important issues, the pollutants that result from the complete life-cycle of various electric generation methods, how the putative hydrogen infrastructure would have to grow if hydrogen were to become a transportation fuel, and other related topics. We also developed a process for estimating levelized costs, including a way of calculating uncertainty without having to use computationally intense Monte Carlo methods. The white papers are summarized in the project's final report, and some have become the focus of reports that are in progress or in printing at this time. Some of this work has enjoyed joint support from other projects.

An idea arose during the conduct of this work for the recycling of $CO_2$ by extracting this greenhouse gas from industrial processes streams and, perhaps, eventually from the atmosphere. Nuclear or renewable energy would be used to separate and recombine the carbon with hydrogen to form synthetic hydrocarbon fuels and other products, closing the cycle for carbon dioxide emissions. The process would also take full advantage of the existing infrastructure for fossil fuel distribution and use. This work provided an avenue for discovery

and raised the prospect of further opportunities to utilize Sandia capabilities.

## Other Communications

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M. Tatro, J.M. Covan, R. Robinett III, G.W. Kuswa, D. Meniccuci, and S.A. Jones, "Toward an Energy Surety Future," Sandia Report SAND2005-6281, Albuquerque, NM, 2005.

# A Mathematical Method for Quantifying the Effectiveness of Management Strategies

*75443*

*J. A. Cooper, J. M. Covan, L. Sena-Henderson, J. D. Brewer*

## Abstract

Large complex teams must achieve sustained productivity in critical operations (e.g., weapons and reactor development) while maintaining safety for involved personnel, the public, and physical assets, as well as security for property and information. This requires informed management decisions that depend on trade-offs of factors such as the extent of personnel protection, potential accident impacts, the degree of information and physical asset protection, and communication with and motivation of involved personnel. All of these interact and potentially interfere with each other and must be weighed against financial resources and implementation time.

Existing risk analysis tools can successfully treat physical response, component failure, and routine human actions. However, many "soft" factors involving human motivation and interaction among weakly related factors remain problematic. There is a need for a more effective software tool for quantifying these trade-offs and helping make rational choices. This type of tool facilitates improvements in safety, security, and productivity, and enables measurement of improvements as a function of resources expended. Operational safety, security, and motivation are significantly influenced by "latent effects," which are pre-occurring influences. One example of these is that an atmosphere of excessive fear can suppress open and frank disclosures, which can in turn hide problems, impede correction, and prevent lessons learned. Another is that a cultural mind-set of commitment, self-responsibility, and passion for an activity is a significant contributor to an activity's success. We investigated an innovative approach for quantitatively analyzing latent effects in order to link the types of factors described above, aggregate available information into quantitative metrics that contribute to strategic management decisions, and measure the results. The approach also evaluated the inherent uncertainties and allowed for tracking dynamics for early response and assessing developing trends. Model development was based on how factors combine and influence other factors in real time and over extended time periods. This proved to be a promising new approach for research and development applied to risk management.

## Accomplishments

We demonstrated several unique and innovative features of the proposed approach:

(1) The analysis structure helps understand the human process, including latent factors such as fear, lack of full disclosure, "brainwashing," and dedicated motivation. This approach solved a previously unsolved problem of estimating likelihood of these types of safety and security failures.

(2) The latent effects decomposition, tied to a mathematical analysis, demonstrated help in understanding processes, interrelating factors, accounting for dependence, and aggregating inputs into meaningful decision aids.

(3) The expert elicitation process blended inputs from operations "owners" with more global information of which they may not have been aware.

(4) Possibilistic uncertainty processing allowed appropriate representations of subjective uncertainty and aggregation of that uncertainty in the latent effects structure. This processing also blended through hybrid analysis with objective data, where available.

(5) The combination of "scoring" risk of various types of problems, along with trends and early alerts, allowed for a realistic global view of productivity, safety, and security impacts.

(6) The business oriented top-down system process for subjecting requirements and responses to cost-benefit processing under normal, degraded, and emergency conditions also proved to have unique value.

(7) We applied the method to problems of varying scale, e.g., a single operation and an entire organization.

(8) We constructed mathematical models applicable to safety-and security-critical operations, including the involved personnel, organizations, and management. We focused the activity on five main thrusts along with some less intense treatment in one or two other areas. The thrusts chosen for emphasis were: personnel performance and evaluation, management per-

formance and evaluation, organization performance, team performance, and organization safety posture.

(9) We decomposed the operation factors into analyzable latent effects structures, chained to show latent effects. In doing this, we gave consideration to game theory, dissipative sustainability analysis, unique signal communication, and various forms of entropy theory.

(10) We developed and tested expert elicitation methods for potential information needs and for the consequences of changes.

(11) We focused on how mathematical inputs are derived from objective data and expert elicitation. Consideration was also given to how outputs can be effectively portrayed.

(12) We developed criteria for judging the degree of success of the project so its value can be assessed based on test cases. This involved validation metrics because we needed to know if the methods developed are effective and useful. Our validation was judged successful.

(13) We developed software for processing the data gathered in a Markov chained latent effects structure with soft aggregation information processing and produced user manuals. Bob Roginski, a retired Sandian, was placed under contract for software development. His experience in implementing the software routines necessary for this approach was valuable.

(14) We constructed several test cases that were used to test model validity. We showed value added, first to our division, second to Sandia, third to the Department of Energy, and fourth to the scientific community in general.

(15) We assessed results of the test cases against the criteria for success. This is the point at which we demonstrated the value of the project. One important metric to this demonstration was the acceptance of the key ideas in three international conference forums and for publication in an international journal.

## Refereed

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## Other Communications

J.A. Cooper, "Flight Control Aging Assessment Using Markov Latent Effects Analysis," presented and in *Proceedings of the Aging Aircraft Conference*, Palm Springs, CA, February 2005, pp. 76–89.

J.A. Cooper, "Management Strategies and Analysis for Safety in High Consequence Systems," presented at the NASA Contractors Safety Forum, Houston, TX, February 2005.

J.A. Cooper, "A Method for Enhanced Estimation of Extreme Hazards," presented at the Warhead Hazardous Environment Protection US-Russian Workshop, Albuquerque, NM, September 2004.

# Inactivation of Avian Influenza (Bird Flu) with Sandia Developed Decontamination Formulations

*76306*

*G. S. Brown, J. M. Bieker*

## Abstract

An avian influenza, influenza A, that infects humans, birds, and swine has been identified in Asia. The avian influenza virus currently affecting Asia has been identified as influenza A hemagglutinin 5 neuraminidase 1 (H5N1). To date, the virus has been detected in avian species in several countries, including Viet Nam, Thailand, and China, with human infections reported in Viet Nam and Thailand. The World Health Organization is recommending affected countries rapidly cull and slaughter H5N1 infected or potentially infected poultry to contain the outbreak. The human cases of H5N1 influenza are believed to be from direct exposure to chickens or chicken waste, with no reported human-to-human transmission at this point. However, containment of the outbreak is essential to mitigate the potential mutation and evolution of the virus into forms readily transmitted from human to human, which could result in widespread illness and death worldwide.

Culling infected flocks alone will not completely contain the outbreak because areas housing the birds contain feces and urine also contaminated by H5N1. Widespread decontamination of all facilities is required to prevent infection of new flocks introduced into these areas and to reduce the threat to humans. Sandia, in collaboration with Kansas State University, previously demonstrated rapid and complete inactivation of bovine corona virus in the presence of high organic loads using Sandia developed decontamination formulations. We proposed a study to evaluate the inactivation effectiveness of the decontamination formulations toward influenza A, which has similar physiochemical properties to corona virus under high organic loading.

## Accomplishments

We completed testing using Sandia developed formulations, such as DF-200 and its modifications, Virkon® S (potassium peroxymonosulfate), and common chemicals such as ethanol and bleach for efficacy against influenza A, including mammalian isolate A/WSN/33 H1N1, swine influenza A H1N1, and low pathogenic avian influenza H5N8. Included in the efficacy testing were 0%, 10%, and 50% organic challenge using poultry fecal and litter material. We determined infectious titer post treatment in Madin-Darby canine kidney cells using endpoint dilution assay (TCID50). Additionally, we determined quantitative ribonucleic acid (RNA) degradation using a real time reverse transcription polymerase chain reaction designed for all influenza A isolates. Results indicate that 100% DF-200 and 10% bleach are the most effective for inactivating influenza A based on both TCID50 and RNA degradation, while 1% Virkon® S and 70% ethanol were much less effective.

# Assessing Geopolitical Instability Caused by Global Climate Change

*79775*

*M. B. Boslough, T. G. Trucano, M. D. Peters, M. A. Taylor, J. A. Sprigg, L. A. Malczynski, L. A. McNamara, G. A. Backus*

## Abstract

Research indicates that global climate trends are impacting the availability of vital resources such as water, food, soil, territory, and stable climate. In recent years, foreign policy experts compiled evidence that such scarcity may precipitate conflict. Understanding the interaction between resource competition and physical Earth systems is critical for anticipating and mitigating conflict, but the mechanisms of coupling are poorly understood. Computational modeling offers a means to develop and test hypotheses about linkages between environmental change and conflict. This three year project will develop an agent model that interfaces with the Community Climate System Model (CCSM) to generate hypotheses about unforeseen thresholds, or "tipping points," caused by environmental stress.

Appropriately, the research team consists of physical, non-physical, and computational scientists. The team will make use of research in the non-physical sciences and in quantitative conflict prediction, including systems dynamics models developed at Sandia and the Center for Army Analysis's Fuzzy Analysis of Statistical Evidence (FASE), to develop a set of qualitative models linking physical and non-physical phenomena. These will form the basis for simple agent based models to explore linkages between physical climate changes and instabilities. Simple agent models, in turn, will be expanded to more complex cellular agent models incorporating a variety of geographical regions, time scales, and macro-structural variables.

Resulting tools will be useful in areas such as intelligence analysis, developing strategies for conflict mitigation, and systems approaches to deploying environmental sensor technologies. However, identifying the mechanisms and creating tools for linking agent based and physical dynamics will be challenging. This project will develop and demonstrate proof-of-principle and preliminary tools.

## Accomplishments

We pursued a multipronged approach to analyze the relationships between climate and conflict and to address the limitations of current conflict-forecasting models. We quantitatively explored causal factors related to the study of state conflict using the FASE method developed by the Center for Army Analysis. We also applied traditional statistical regression methods. The FASE pattern classification algorithm provided conditional probabilities of conflict, based on historical data, for such independent variables as youth bulge, infant mortality rate, and trade openness. We also extended the data set to incorporate the occurrence of natural disasters.

In our causal factors study, we found that there are significant relationships between various conditions of a region and its likelihood to engage in conflict. With regard to climate change, we found that food consumption and casualties from natural disasters can affect conflict. For example, climate change could decrease crop yields or increase the likelihood of disasters like flooding and tidal waves, thereby increasing the instability and likelihood of conflict. We identified case study scenarios (Milestone #1). We monitored several emerging conflicts in the world, including Bangladesh and the Sudanese region of Darfur. We selected the Darfur conflict as a candidate case study for further analysis. We reviewed and tested other statistical methods from the behavioral and social sciences to further probe the causal relationships between climate and conflict. These methods are qualitative choice theory, cointegration, Granger causality, and Bayesian causal discovery. We identified the process that would be required to couple an agent-based model with a climate model. Finally, we reviewed agent models, including a survey of open source software (Milestones #2 and #3). We chose the Repast open source toolkit, as well as the AnyLogic commercial package for year two prototyping and agent-based model development.

# Nanoscale Optical and Electrical Probes of Materials and Processes

*79776*

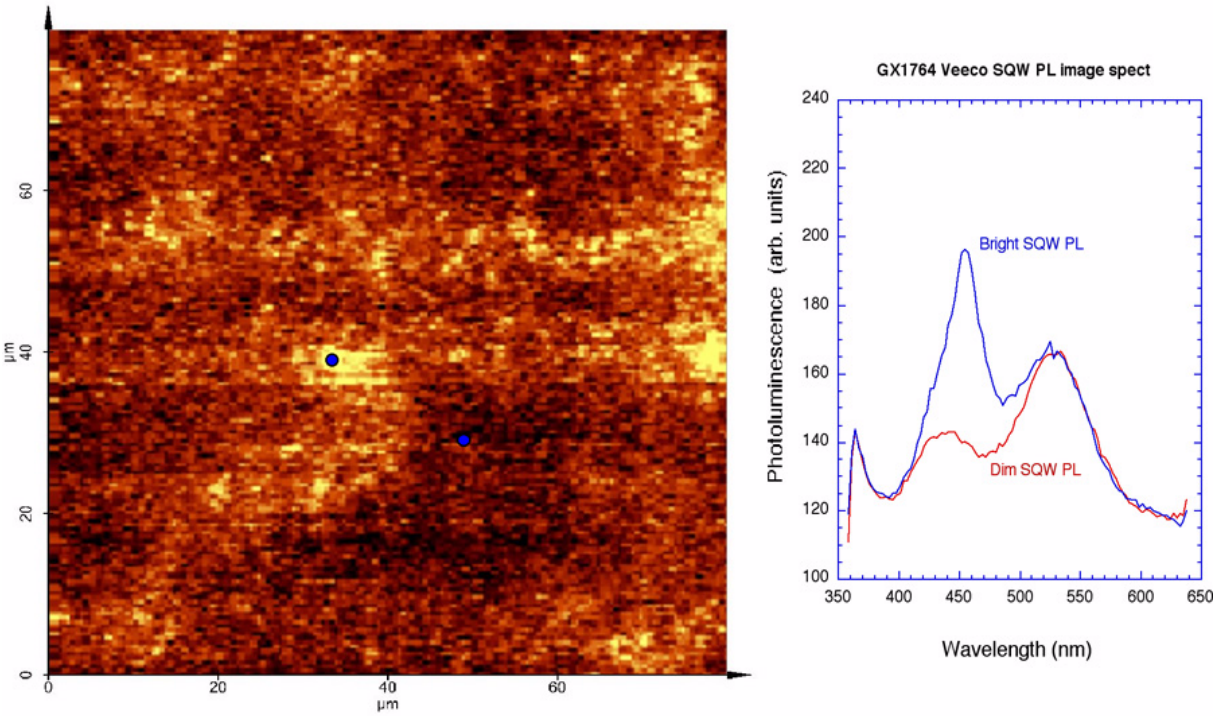
*K. H. Bogart, M. A. Quintana, S. R. Kurtz*


## Abstract

Near-field scanning optical microscopy (NSOM) integrates optical and scanning probe microscopy (SPM) techniques that generate simultaneous optical, electrical, and structural images with nanometer scale resolution. We are developing new measurements with a state-of-the-art NSOM-SPM instrument that is unique in its ability to concurrently capture electrical, morphological, and spectrally-resolved optical information spanning the 325–1100 nm wavelength range. These capabilities are ideal for investigating materials and processes associated with solid-state lighting (SSL) and solar technologies and will enable us to provide direction to materials growth and device processing activities as well as assist in the development of more efficient light emitting diodes (LEDs) and solar cells.

Work in FY 2005 focused on utilizing the NSOM system to identify issues that influence InGaN/GaN LED output power and efficiency. We found initial LED emission occurred close to the n-contact, indicating a significant voltage drop to the underlying n-type contact layer. Using confocal microscopy (~1 µm resolution), we determined that emission, p-n junction photocurrent, and surface morphology all displayed the same features spatially, and that light input/output is dominated by scattering from large surface features such as hillocks and V-defects. We compared confocal photoluminescence images of single quantum well (SQW) InGaN/GaN materials from Sandia and Veeco (formerly Emcore). Higher resolution (= 0.1 µm) photoluminescence imaging with the NSOM is currently under development. We added a photomultiplier to increase detection sensitivity over the charge coupled device (CCD) array, but the NSOM of our current Sandia InGaN/GaN SQW samples remains at our sensitivity limit.

We performed initial scanning probe experiments with conductive tips and devoted significant time to


*SQW photoluminescence intensity integrated over 420–470 nm. Yellow-black corresponds to 94-19 relative intensity range. Spectra: Bright and dim SQW photoluminescence spectra obtained at two points in the image indicated in blue.*

configuring the experiments to accommodate a large tip-surface impedance. We performed initial cross-sectional mapping of the CdTe solar cell band bending. Consistent with the cell design, the width at the open circuit of the depletion region was comparable to our resolution (0.1 µm), and the potential image consisted of a sharp step.

## Accomplishments

Work with the NSOM system during FY 2005 focused on utilizing the instrument to identify issues that influence nitride LED output power and efficiency.

With confocal microscopy, we compared LED emission, p-n junction photocurrent, and surface morphology (monitored with laser reflectance). Comparing images with < 1 µm resolution, we find that emission, photocurrent, and surface morphology all display the same features. In the latest generation of Sandia LEDs, light output/input is dominated by scattering off of large surface features like hillocks and V-defects.

We compared confocal photoluminescence images of SQW LED material from Sandia and Veeco/Emcore. Unlike the emission and photocurrent, the photoluminescence displayed micrometer-sized bright regions that were not associated with surface features. Photoluminescence images and representative point spectra for a state-of-the-art Veeco wafer showed a significant fraction (~50%) of the 420–470 nm SQW emission originated from the bright (yellow) regions. The longer wavelength, 480–580 nm GaN "yellow-band" occurred more uniformly throughout the wafer. The SQW peak is shifted ~15 nm to longer wavelength, corresponding to a 1–2% higher indium content in the bright regions. These data indicate that indium segregation is present in these films and can be detected with our instrument.

Although initial results are encouraging, we need to examine more samples in order to understand indium segregation. With our new nitride metal-organic chemical vapor deposition (MOCVD) reactor, we expect that the Sandia material will continue to evolve and demonstrate increasing photoluminescence and LED brightness. With emission occurring preferentially from micron-sized domains, we plan to continue to investigate the nature of these regions and the growth conditions controlling their formation. We designed experiments to correlate specific InGaN MOCVD growth parameters with indium domain formation and emission characteristics. If we understand the effects and can control indium domain formation, we can optimize InGaN materials for LEDs used in solid-state lighting. Our instrument is proving to be the best tool to distinguish indium segregation from defect/dislocation models as competing explanations for the formation of emission domains.

Higher resolution (< 0.1 µm) photoluminescence imaging with the NSOM is under development. We added a photomultiplier to increase detection sensitivity over the CCD array. Even with this upgrade, SQW NSOM remains at the limit of our sensitivity, and we are not able to produce publication quality NSOM images of the SQW samples examined so far. Improved brightness in InGaN materials will increase our ability to detect signal in the high-resolution NSOM mode and understand emission from these domains. We are also working with the microscope vendor to upgrade the NSOM system and improve optical detection sensitivity.

Initial scanning probe experiments with a conductive tip were performed. We devoted substantial time to configuring the experiments to accommodate a large tip-surface impedance. We performed initial scanning probe experiments to map the band-bending of a CdTe solar cell cross section. Consistent with the cell design, the width at the open circuit of the depletion region was comparable to our resolution (~0.1 µm), and the potential image consisted of a sharp step. Sample preparation issues arose as the metal contact on the solar cell appeared to smear into the junction region during the cross-sectional cutting procedure. Experiments to optimize sample preparation and limit structural damage to the solar cells are under way. We obtained CdInSe2 (CIS) solar cells for cross-sectional scanning probe potential mapping experiments. There is considerable interest in the CIS technology because of its relatively high conversion efficiency.

# Applying New Network Security Technologies to SCADA Systems

*79777*

*J. E. Stamp, S. A. Hurd, B. J. Smith, M. J. Berg, J. Margulies*

## Abstract

Supervisory Control and Data Acquisition (SCADA) systems for automation are very important for critical infrastructure and manufacturing operations. SCADA systems collect and transmit information between sensors, controllers, and central management stations; in the process, they store, process, and analyze information. They have been implemented in a number of physical environments using a variety of hardware, software, networking protocols, and communications technologies.

One common thread among SCADA systems is that 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. However, even for contemporary SCADA technology, the most important factors appear to be (1) the lack of a perceived business case for SCADA security features, and (2) the reluctance of SCADA operators to implement security features that might impede the operation of the SCADA system.

Much research at Sandia and elsewhere focused on how to improve security and reliability for next generation SCADA systems over the long term. However, there is a critical need to identify ways to address security shortcomings in the short and medium term.

Our project will investigate how technologies developed to secure conventional information technology (IT) networks could be applied to securing SCADA systems and networks. This project's end goal is to identify and test technologies that will significantly improve the security posture of existing SCADA systems. These proposed solutions must be relatively simple to implement, reliable, and acceptable to SCADA owners and operators.

## Accomplishments

We completed the process of identifying a representative sample/model for SCADA systems. We received feedback on our representative sample/model work from industry and integrated their feedback into a relevant report. We began analysis of the *TSWG Guidebook of SCADA Best Practices* to leverage and incorporate information from that document.

We developed an initial report of methods used to secure traditional IT systems and networks and assembled an expert team to integrate security into SCADA systems. We are converting technical best practices, along with other promising techniques to secure traditional IT systems, into our initial security architecture for SCADA.

We engaged student interns from the Center for Cyber Defenders to build a general purpose security appliance (in the form of a Linux based computer). The device secures automation systems either on a per-host or per-network basis. It supplies security services to compensate for poor security built into SCADA components. It encrypts packet traffic, encapsulates and encrypts serial traffic, and serves as a secure gateway for remote SCADA maintenance. It also works as an intrusion detection system and acts as a sensor for active SCADA network visualization software (to analyze in real time SCADA-specific security conditions).The design has won very favorable feedback from industry. The team purchased SCADA software and hardware to support testing plans for FY 2006.

Finally, we identified the development of a SCADA virtual environment as key for FY 2006 testing of the comprehensive SCADA security architecture, and began assembling the requisite team to develop this novel technology.

## Other Communications

M.J. Berg and J.E. Stamp, "A Reference Model for Control and Automation Systems in Electric Power," presented and in *Proceedings of the 2005 Clemson Power Systems Conference*, Clemson, SC, March 2005, CD-ROM.

# Use of Composite Materials to Refurbish Our Civil and Military Infrastructure

*79778*

*D. P. Roach, J. L. Novak, K. B. Pfeifer, D. C. Hammerand*

## Abstract

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 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 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 issues are inherent. This material 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 2003 National Bridge Inventory Database indicates that 30% of the 591,000 bridges in the United States are "structurally deficient."

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, testing of prototype installations was coupled with theoretical modeling in the following four element approach: (1) design and performance of composite repairs, (2) in situ sensors for real time health monitoring, (3) self-healing of in-service damage in a repair, and (4) numerical modeling.

## Accomplishments

- *Design, Installation, and Performance Assessment of Composite Repairs*

Eight different mechanical and chemical surface preparation processes were evaluated for bonding composite materials to steel structures. We completed wedge (crack growth), lap shear (fatigue and ultimate strength), and peel tests on pristine specimens and specimens conditioned in a worst case environment to arrive at an optimum process for installing composite repairs. In addition to being robust, the selected installation process is inexpensive and uses readily available equipment and chemicals. Methods and necessary equipment were proven for high temperature and vacuum bag cures in the field. Initial performance tests established the viability of composite doubler repairs to reinforce steel structures and minimize or eliminate the effects of cracks, corrosion, or other flaws.

- *Smart Structures and Health Monitoring Sensors*

By positioning sensors around the critical regions of the doubler, it is possible to establish an in situ network for continuous, real time health monitoring. In FY 2005, we produced four different sensor designs for our study: (1) fiber optic Bragg grating sensor, (2) micromirror fiber optic sensor, (3) piezoelectric transducer, and (4) high frequency eddy current sensor. We produced innovative multi-axis fiber optic Bragg grating (FOBG) sensors with both extensional and lateral strain sensitivity. We installed FOBG sensor systems on two composite doubler repair specimens to detect the onset of any flaws within the footprint of the repair. Subsequent fatigue tests established the ability of this sensor array to detect crack growth as well as the presence of disbond flaws in the repair. We also accurately measured strain levels and validated the measurements by strain gauges. We also developed a fiber optic micromirror sensor. This sensor allows for the production of a low cost monitor that is ideal for permanent monitoring of crack propagation past a boundary. Tests clearly demonstrated that if a crack propagates across the fiber, which is rigidly connected to the structure

under test, a breakage occurs and is observed at the second optical detector.

We built piezoelectric transducers (PZTs) by embedding PZT materials in a thin dielectric carrier film. We designed the PZT sensor network to interpret signal anomalies for damage detection and location. Fatigue tests showed that these sensors can detect both crack and disbond flaws. Triangulation methods allow for accurate flaw placement. We developed two-dimensional imaging and algorithms to accurately track crack length during propagation. Another subtask investigated high frequency eddy current (EC) sensors to detect cracks, delaminations, and weak bonds. We produced and tested several EC sensor prototypes. We achieved successful crack detection with a dual coil configuration that combines a pancake excitation inductor with a co-located pickup coil to produce a transducer that requires very little drive current.

- *Self-Healing in the Composite Laminate Repair*

Tasks completed to develop the self-healing material specifications include: (1) identifying materials and requirements for the composite doubler adhesive system, (2) surveying potential failure modes for adhesives and identifying self-healing strategies, (3) exploring ring-opening metathesis polymerization based healing agents for adhesive systems, and (4) conducting performance studies of candidate self-healing material systems. The first set of tests produced healing efficiencies (strength) ranging from 52% to 92% depending on the conditions.

- *Numerical Modeling*

We developed and analyzed a number of finite element models of the composite doubler repaired steel plates. The models correspond to thick steel plates with and without doubler repairs and with and without degraded properties in the repair (e.g., poor welds, adhesive disbonds). We conducted initial explorations into the design space by performing computational simulations of models with thicker adhesive layers and steeper doubler taper ratios. Post-processing of the results includes contour plots of stresses and strains, the overall load carried by the plate, and the volume averaged stresses in each layer. By including models with and without the composite doubler present, it is possible to study the overall effectiveness of each composite repair design feature.

## Refereed

D.P. Roach and N.J. Goldfine, “Corrosion and Fatigue Monitoring Sensor Networks,” presented and in *Proceedings of the 2005 Stanford Structural Health Monitoring Workshop*, Palo Alto, CA, September 2005, vol. 1, p. 8.

D.P. Roach, K.A. Rackow, and P.D. Walkington, “Development of Bonded Composite Doublers for the Repair of Oil Recovery Equipment,” Sandia Report SAND2005-3195, Albuquerque, NM, 2005.

# Desalination Utilizing Clathrate Hydrates

*79779*

*B. A. Simmons, R. W. Bradshaw, R. T. Cygan, D. E. Dedrick, E. H. Majzoub, J. A. Greathouse*

## Abstract

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 or reverse osmosis, 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.

Clathrate hydrates have been previously investigated as a means to desalinate water with mixed results. We encountered major obstacles, including identifying the appropriate hydrate formers and efficiently removing excess salt left from the original solutes and/or entrapped in the formed hydrate. We are investigating a novel approach that may solve these problems. The approach uses novel hydrate formers and a bulk inert heat exchange liquid. The heat exchange liquid utilizes the different physical properties of the formed crystals (hydrate and solute) and the chemical species used in the nucleation process. This results 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 utilizing 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.

## Accomplishments

This project met and exceeded all FY 2005 milestones. We started by developing, building, and utilizing a commercially available system that allows for batch desalination. We accomplished this by fabricating and/or modifying two existing reactors. The reactors 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. The results obtained are in good agreement with the data published on these systems.

Once the reactors proved effective in the basic formation and growth of stable hydrates, we conducted initial desalination experiments. We formed the hydrates in a 1.80 wt% salt solution using ethylene as the hydrate forming species. After we observed hydrate formation, we removed the hydrates from the test cell. We melted the hydrates and compared the salinity of the resultant water to that of the residual concentrated and the initial saline solutions. The melted hydrate phase hydrate contained 0.37 wt% NaCl compared to 1.80 wt% in the starting solution and 2.13 wt% in the brine after the test, producing a removal efficiency of 79.4% in a batch process without washing. We already began developing the next generation process that will include a heat exchange liquid that will simultaneously solubilize the hydrate forming species to aid in the hydrate nucleation and growth process and provide a medium in which the buoyant separation of the hydrates from the remaining concentrated salts and brine occurs. We also made significant progress in the computational modeling of multiple hydrate types, including those that have methane as the hydrate forming species.

## Other Communications

J.A. Greathouse, “Atomistic Simulations of Gas Hydrates: Application to Water Desalination,” presented at the Mesilla Workshop, Las Cruces, NM, April 2005.

# Development and Application of the Dynamic System Doctor to Nuclear Reactor Probabilistic Risk Assessments

*79780*

*S. Dunagan, D. M. Kunsman*

## Abstract

Probabilistic Risk Assessments (PRAs) of nuclear reactors are notoriously resource intensive. They are increasingly relied upon by the US Nuclear Regulatory Commission (NRC) in making licensing decisions for current and advanced reactors. Yet PRAs are produced much as they were 20 years ago. Our idea applies one modern systems analysis technique to a specific part of the PRA: the accident progression event tree (APET).

The Dynamic System Doctor (DSD) is system independent, interactive software under development at Ohio State University (OSU) for model based state/parameter estimation in dynamic systems. It can provide input to real-time analysis for evolving systems conditions and has been successfully applied to real-time xenon estimation in a reactor, in stability analysis for a boiling water reactor, and in fault detection in automobile engines. The DSD has also been linked with a neural net approach.

Our idea links DSD to MELCOR so that it can directly and semi-automatically help us construct additional tree logic. MELCOR is a detailed thermal/hydraulic severe accident code developed at Sandia. We originally thought that the DSD would link directly to the APET that then would link to the MELCOR model. We found that it is more efficient to link the driver directly to MELCOR, from the perspective of an overall PRA. Furthermore, an integrated model could be utilized in reactor control rooms to provide operators with real time severe accident progression to help them mitigate such an accident. Our chosen problem is modeling a pressurizer. Its conditions impact the thermal hydraulics of the coolant system throughout the accident and hence affect the entire APET logic.

## Accomplishments

Original Phase I project milestones were the following:

(1) Develop multi-tasking driver (MTD) for dynamic event tree generation.

(2) Develop reduced-order model for steam generator.

(3) Link the driver and the DSD for reduced-order model.

(4) Compile the database for the event-tree branching probabilities.

Work toward Phase I milestones progressed simultaneously. In particular, work toward Milestones 1 and 3 is proceeding as a single task upon the recommendation of the professor at OSU who is our parallel processing and distributed computing expert. The reduced order model we will use toward Milestone 3 is a pressurizer model (rather than a steam-generator model).

- *Milestones 1 and 3:* We developed a prototype MTD in Java that works on Windows, Linux, and Mac OS X). The Java driver was successfully linked and tested with the pressurizer model. Now we will start to develop a more general driver we will develop using C/C++. We will use a framework that will provide the infrastructure for multi-threaded execution on a variety of hardware architectures, such as single and multi-processor machines, homogenous and heterogeneous clusters, and distributed environments. The C++ driver will link MELCOR.

We identified the interface points with MELCOR. The MELCOR input files will be modified by the driver code, and with the help of control functions, we will stop the simulation at branching points. The driver will modify the input files to follow the branches, and it will execute MELCOR with the new input files. We are currently developing a machine readable format for editing rules for the input files. We will generate the file we call “Edit-Rules,” using the user-input branching rules. It will be used by the driver to modify the input files at the branching points.

- *Milestone 2:* Completed (with pressurizer model).
- *Milestone 4:* We identified key events toward the selection of the branching and pruning rules for the test problem (loss of all AC power compounded by a loss of all feedwater to the steam generators). The test case will be simulated by the MELCOR code. The example plant under consideration is Zion. We acquired and installed both the MELCOR code and the Zion input deck for MELCOR.

We also made substantial progress toward two Phase II milestones: linking the driver and DSD to an actual steam-generator model and developing output graphics.

# Innovative Solar Thermochemical Water Splitting

*79781*

*R. B. Diver Jr., J. E. Miller, R. E. Hogan Jr., M. D. Allendorf*

## Abstract

We are investigating 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 therefore investigated ferrite thermochemical processes in the context of a Sandia invention that utilizes two sets of moving beds of ferrites in close proximity to provide radiation driven, countercurrent heat recuperation. This invention provides inherent separation of the product hydrogen and oxygen, is conceptually simple, and is an excellent match with high concentration solar thermal technology. A system performance model, using the thermodynamics of iron oxide and mixed metal ferrites with detailed thermal models, indicates that temperature requirements can be reduced substantially compared to iron oxide alone. It also indicates that high thermal efficiency is possible. However, simulations indicate that temperatures of over 1800 K are still required. Therefore, we experimentally and theoretically investigated various combinations of mixed metal ferrite, with use of zirconia support, to tailor the thermodynamics to disperse the ferrite and improve its reactivity. Preliminary results indicate that the zirconia support helps avoid slag formation issues and provides oxygen transport to and from the redox sites. If our planned feasibility tests can demonstrate that mechanical and chemical stability of ferrite impregnated zirconia monoliths can be maintained, the potential for distributed competitive solar thermochemical hydrogen production is good.

## Accomplishments

The first year of the project had two major thrusts. The first was to develop system models that simulate the key parameters in the proposed design. Because of the modeling complexity inherent in the design, including radiation heat transfer between moving parts, multispecies flow, and chemical reactions, we focused our model development primarily on radiation driven recuperation and buoyancy driven flow. We deferred a detailed coupled analysis, in which all key mechanisms are simultaneously solved. The results support previous conclusions that high recuperation efficiency is possible and suggest that buoyancy driven mixing between the two sections of the reactor takes place, but can be controlled.

The second major thrust was related to developing materials that meet the goals outlined by the systems models. To understand metal oxide redox thermodynamics, we used commercially available thermodynamic databases such as HSC Chemistry for Windows and FactSage. FactSage, in particular, was most useful in modeling solid solutions of iron oxides and other metal oxides in stabilized zirconia. We established an experimental capability for cyclically heating metal oxide samples, and small amounts of thermochemical hydrogen were produced. Our initial results with nickel manganese ferrite were comparable to the best results reported in the literature. An important result from our material work is an improved appreciation of the zirconia support. Thermodynamically, it appears that it enhances oxygen production during the reduction reaction. It may also permit operation at temperatures higher than previously considered practical by providing support for iron and other metal oxides that normally melt near the temperatures of interest, while holding some of the metal oxide in solid solution. In addition, as an excellent oxygen conductor, particularly at high temperatures, it appears that reaction/transport kinetics may be exceptionally high and that maintaining high porosity and associated sintering issues may not be an issue.

# Ultraviolet Water Purification Systems for Rural Environments and Mobile Applications

*79782*

*M. H. Crawford, M. P. Ross, M. A. Quintana, A. A. Allerman, R. Boucher, D. S. Ruby*

## Abstract

The recent demonstration by Sandia researchers of solid state ultraviolet (UV) light emitting diodes (LEDs) in the germicide wavelength range (230–300 nm) will enable the development of low-cost, sustainable water purification systems for rural environments and mobile point-of-use applications. Clean, safe, potable water is an ever increasing issue worldwide, with significant impacts on national security. By combining low power consuming solid-state devices with photovoltaic systems designed to take advantage of the unique properties of LEDs (rugged, long lifetimes, low power), this LDRD seeks to develop a "leap frog" technology platform in the area of biological decontamination of remote and mobile water sources. This project is focused on establishing baseline biological studies of UV inactivation processes.

Our development efforts have produced an LED based water exposure setup with uniform beam spatial profile and made advances in deep UV LED performance. We demonstrated *Escherichia coli (E. coli)* inactivation with 270–300 nm LEDs and assessed the potential for applying deep ultraviolet LED based water purification to mobile point-of-use applications as well as to rural and international environments where the benefits of photovoltaic-powered systems can be realized.

## Accomplishments

This project had three primary technical goals: (1) Perform UV inactivation studies using Sandia developed deep UV LEDs; (2) perform an assessment of rural and international UV water purification systems including design considerations for an LED based system; and (3) perform an assessment of mobile point-of-use UV purification systems including design considerations for an LED based system.

Our first milestone for the UV inactivation studies required completing the design, assembly, and characterization of an optical test setup to deliver relatively uniform and collimated UV light from UV LED arrays with, normally, very non-uniform and divergent beam properties. We accomplished this milestone through the integration of a 2x2 element deep UV LED array, with custom reflectorized packaging, into a beam collimator. The assembly consists of a six inch long aluminum tube with a textured internal surface. We met our second milestone through the completion of germicidal inactivation measurements using this LED apparatus as the excitation source. Our studies evaluated the impact of LED wavelength and incident dose on the inactivation of various strains of *E. coli*, a common contaminant in water samples. Through development efforts, our LED performance improved significantly throughout the project. Our first LED sources delivered 20 microwatts at 272 nm, followed by a 295 nm source with 100 microwatts of power, and finally, a 270 nm source providing 170 microwatts of power (all measured at the exit of the beam collimation tube and including optical losses from that system). For the 295 nm source, we completed *E. coli* inactivation studies as a function of incident LED power and found that an incident dose of 78 mJ/cm$^2$ enabled 99.99% inactivation of *E. coli* strain ATCC 11229. With the improved 270 nm source, we investigated a different strain of *E. coli*, ATCC 15597, and determined that an incident dose of 264 mJ/cm$^2$ enabled 99% inactivation. Transmission studies on the *E. coli* ATCC 15597 samples determined that the sample absorption at 270 nm was approximately 1.3% in our 190 microliter sample volumes, yielding a total absorbed dose of 3.4 mJ/cm$^2$. This dose is within a factor of two of published values for 99% inactivation using traditional mercury lamp excitation. It also demonstrates the effective utilization of deep UV LEDs for *E. coli* inactivation in water samples.

To meet the second and third goals of the project, we researched two important application areas for UV water purification where LEDs may have a distinct advantage. We determined that certain system performance advantages are especially important for water purification systems that will be used in rural areas of developing parts of the world. Where grid electricity is not available, safe drinking water is hard to come by, infrastructure for replacement parts is far away, and general expertise is not common. Advantages of deep

UV LEDs in these applications include the anticipated longer lifetimes compared to traditional low power mercury lamps and the ability to include more frequent on-off cycles in the overall designs. These benefits translate to lower life cycle costs, more reliable designs, and the ability to use intermittent energy sources—all of which contribute to the overall sustainability of the complete water purification solution (purifier plus energy source). We further examined the requirements of UV water purification systems in mobile point-of-use applications where no external energy source is available. We specifically considered photovoltaically powered systems. Key advantages of LEDs for this application include potential for longer lifetimes, lighter weight, lower cost, greater robustness, greater possibility for integration and flexibility in system design, and faster turn-on and power stabilization times.

# Physical Modeling of Scaled Water Distribution System Networks

*79797*

*T. J. O'Hern, S. A. McKenna, B. G. van Bloemen Waanders, L. Orear Jr.*

## Abstract

Threats to water distribution systems include release of contaminants and Denial of Service (DoS) attacks. 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. Validation data are needed to evaluate numerical models of network operations. Some data can be acquired in real world tests, but these are limited by (1) unknown demand, (2) lack of repeatability, (3) too many sources of uncertainty (demand, friction factors, etc.), and (4) expense. In addition, real world tests have limited numbers of network access points.

We fabricated a scale model water distribution system and acquired validation data over a range of flow (demand) conditions. Standard operating variables included system layout, demand at various nodes in the system, and pressure drop across various pipe sections. In addition, we varied the location of contaminant (salt or dye) introduction. Measurements of pressure, flow rate, and concentration at a large number of points, and overall visualization of dye transport through the flow network were completed. Scale-up issues incorporated in the experiment design include Reynolds number, pressure drop across nodes, and pipe friction and roughness. We chose a 20:1 scale, so we modeled the 10 inch main with a 0.5 inch pipe in the physical model. We ran controlled validation tracer tests to validate flow and transport models and to validate the degree of mixing at pipe junctions. Results of the pipe mixing experiments showed large deviations from predicted behavior, and these have a large impact on standard network operations models.

## Accomplishments

This project focused on demonstrating the feasibility of using a scale model to provide validation data for computational models of water distribution networks.

(1) We performed simulations supporting the experiment design. These started with a simple pipe "cross" junction and were extended to a small pipe network. The simulation results guided the experiments in terms of where measurements were needed and what range of flow conditions should be covered.

(2) We completed single junction experiments. Data showed incomplete mixing at cross junctions. This result shows a weakness in the numerical models that assume complete mixing at every junction.

(3) We built and tested the pipe network. This 20:1 scale model was fabricated with clear PVC pipe to allow optical measurements of, for example, dye transport and mixing in the pipe network. Much of this work was performed by students at the University of New Mexico.

(4) We acquired pressure, flow, and mixing data in the pipe network.

(5) We created a computational model of the network, but it has not yet been validated.

# System Analysis of Carbon Sequestration with Clean Coal Technology

*79798*

*P. H. Kobos, L. E. Klebanoff, O. H. Paananen*

## Abstract

Carbon sequestration is the capture and storage of $CO_2$ from the combustion of fossil fuels and biomass. Increasingly, carbon sequestration is an option to support the transition in energy production and reductions in greenhouse gas emissions over the next 50 to 100 years. Recognizing the role of carbon sequestration, the Administration has incorporated an increase in funding for carbon sequestration research in its budgets. The Department of Energy recently released a long term plan to develop clean coal technology as part of its strategy to turn coal into gas for hydrogen fuel cells and to capture and store up to a million tons of $CO_2$ per year.

Several issues arise in assessing whether a clean coal technology and carbon sequestration strategy represents a viable option to support future energy needs. These issues include understanding the life cycle costs for a clean coal energy system with carbon sequestration, the actual capacities for carbon separation and storage, and the technology and infrastructure requirements to enable economically feasible carbon separation and sequestration in a clean coal energy system.

To address these issues, this project develops a summary of clean coal and carbon sequestration technology systems and costs from the literature, including sequestration capabilities and new technology and infrastructure components. This project also identifies where the systems analysis results could play a role in current and potential Sandia energy models. The main findings illustrate there may be very large potential for $CO_2$ sequestration throughout the world and, at this point in time, the largest component of a sequestration system's cost (e.g., power plant, $CO_2$ capture technology, $CO_2$ transportation and $CO_2$ disposal in a sink) may lie with the technology used to capture the $CO_2$.

## Accomplishments

The accomplishments of this project include addressing the technical, economic, and systems-oriented questions regarding clean coal and carbon sequestration technologies. The project investigated the following specific areas:

(1) Addressing the technical rationale for uses of $CO_2$ and how they relate to large scale carbon sequestration systems.

(2) Characterizing coal based power systems with respect to their designs and potential to remove $CO_2$ from their flue gas where possible. The project characterized coal-fired boiler-based plants, integrated gasification combined cycle (IGCC) plants compared to pulverized coal (PC) plants, fluidized bed combustion systems, pressurized fluidized bed combustion systems, and ultra supercritical pulverized coal with respect to $CO_2$ output, $CO_2$ capture potential, and the related system economics.

(3) Addressing full systems costs and technologies by investigating the $CO_2$ removal methods, transportation, and disposal (or use) potential in the US and worldwide.

(4) Identifying potential barriers and opportunities for future $CO_2$ sequestration markets and projects.

We succeeded in characterizing existing carbon capture and sequestration technologies, costs, and systems at a high level. With this basis of understanding, current or future energy models at Sandia that could build upon this information include the Hydrogen Futures Simulation Model, the Electricity Generation Cost Simulation Model (GenSim), and other models where appropriate. Our findings point to relatively high carbon sequestration costs, especially with respect to capturing the carbon at the source. New technology and advanced systems may enable costs to decrease over time, enabling projects to couple the advanced coal burning power plants with the comparatively inexpensive $CO_2$ transportation and sequestration infrastructure. Additionally, there is a relatively large potential for $CO_2$ sink capacity (decades to centuries or longer) throughout the world.

## Other Communications

D.J. Borns, O.H. Paananen, P.H. Kobos, and L.E. Klebanoff, "Carbon Sequestration and Clean Coal Technologies: Characterizing Systems and Evaluating Costs," Sandia Report SAND2005-5917P, Albuquerque, NM, 2005.

# MOCVD Synthesis of III-Nitride Heterostructure Nanowires for Solid State Lighting

*79800*
*G. T. Wang, J. R. Creighton*

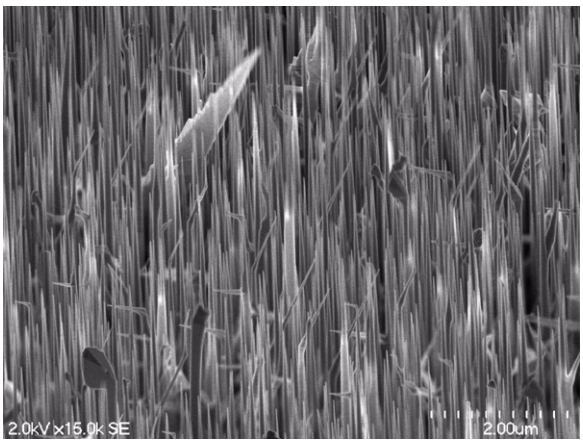
## Abstract

This project deals with the development of scalable metal-organic chemical vapor deposition (MOCVD) based processes to synthesize and characterize single-crystalline III-nitride nanowires and heterostructure nanowires for potential novel solid-state lighting and photonic nanodevice applications. Our approach focuses on using MOCVD to synthesize the nanowires 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 light emitting diodes (LEDs) and solid-state lasers. We believe that success in developing MOCVD based techniques for controllably synthesizing III-nitride nanowires would represent a major advance in nanowire device research and 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.

## Accomplishments

In order to develop our technique for synthesizing III-nitride nanowires, we built off of our prior and continuing experience in the growth of GaN nanowires via MOCVD. The GaN nanowires serve as the core upon which shell layers of different III nitride materials can be heteroepitaxially deposited. We achieved this by varying the process conditions to quench axial nanowire growth and to allow the new layer to grow radially to form a shell around the GaN nanowire core. In this manner, we synthesized core-shell nanowires with AlN, AlGaN, and InGaN shell layers on 2 inch sapphire substrates. We also performed preliminary experiments fabricating more complex three and five layer superlattice nanowires using this technique. However, accurate structural and compositional characterization of the heterostructures nanowires proved difficult. We are currently developing and refining techniques to determine the uniformity (both within a nanowire and across nanowires on the same sample), thickness, and quality of the heterostructure layers. This will allow us to better refine our growth processes.

We also made significant progress on the alignment of III-nitride nanowires. Under specific process conditions, we were able to grow dense, extremely well aligned nanowire arrays on sapphire without the use of a template or patterning. Developing techniques for growing aligned arrays of nanowires is of significant interest, as it may be required for applications requiring anisotropy, including light extraction and lasing. Well ordered nanowire arrays may also facilitate more uniform growth of the heteroepitaxial shell layers during growth of the heterostructure nanowires. We also performed preliminary experiments to intentionally n-type dope GaN nanowires using silane gas.




*Highly aligned vertical array of GaN nanowires grown by MOCVD.*

# Novel System for Zero Emission Electricity and Hydrogen Production from Coal and Biomass

*79801*

*C. R. Shaddix, R. W. Schefer, J. C. Oefelein, A. E. Lutz, T. C. Williams*

## Abstract

Coal is one of the most abundant, low cost energy resources in the US, currently supplying half of our electric power generation. Consequently, coal will play a significant role in US energy security for the foreseeable future. Biomass is a form of renewable energy that is projected to become an increasingly important contributor to the US energy supply. Demonstration projects show that through integrated gasification combined cycle (IGCC) technology, both of these fuels may be used to generate electric power with very low emissions of traditional pollutants and good thermal cycle efficiencies. However, interest in the use of hydrogen as a pollution-free energy carrier is motivating the analysis of prospective, low cost means of generating large quantities of hydrogen. Coal and biomass gasifier systems are promising sources of hydrogen when coupled to the production of a high purity stream of $CO_2$ suitable for sequestration or other end use. Existing analyses of such systems have focused on maximizing $H_2$ production, resulting in the neglect of systems that optimize the fuel conversion system for combined production of power and hydrogen. These analyses also neglected practical limitations in the gasification and gas turbine combustion processes that impact realistic system operation. This project, in collaboration with researchers at the Department of Energy's National Energy Technology Laboratory (NETL), focuses on making new and important measurements of the gasification process and of dilute oxy-fuel firing of $H_2$-depleted syngas in a model gas turbine combustor. The combustor results are guiding and validating a large eddy simulation (LES) model of the combustor that predicts the performance of a practical gas turbine combustor. We are incorporating the results of the gasifier and combustor investigations into a systems simulation model to identify the optimal performance characteristics of this system.

## Accomplishments

We achieved significant progress in FY 2005. We held a project kick-off meeting at the NETL site in Morgantown, WV. During this meeting, we identified areas of Sandia/NETL collaboration, including experimental measurements and advanced modeling of gas turbine combustors and Simulink systems analysis of coal based energy systems for coproduction of power and hydrogen. To conduct the appropriate gas turbine combustor experiments at Sandia, modifications were necessary to the laboratory flow handling and control system to allow the use of a mixture of oxygen and carbon dioxide as the burner oxidizer (instead of air). We completed these modifications, including the reprogramming of the burner control software. In addition, we installed an improved gas turbine combustor simulator. Initial results suggest that the new burner operates well, and stable flames are readily achievable for methane or syngas mixtures burning in air or synthetic oxidizers composed of dilute oxygen. A literature review revealed that the effects of gasifier temperature and pressure on hydrogen production have not been closely examined. The literature does suggest that for high temperature entrained flow gasifiers, the composition of the product gas mixture can be predicted quite accurately through the use of equilibrium calculations. This implies that one may predict the gasification conditions that optimize the yield of hydrogen and suggests that an equilibrium calculation, together with a char gasification rate calculation, is suitable for modeling the gasifier performance in a system model. We added a number of new, first generation component modules to the Simulink coproduction system model, including an air separation unit, gasifier, membrane separator, gas turbine, and heat recovery steam generator combined with a steam turbine.

# Fuel Traps: Mapping Stability via Water Association

*79803*

*S. L. Rempe, R. T. Cygan, K. Leung, T. M. Alam*

## Abstract

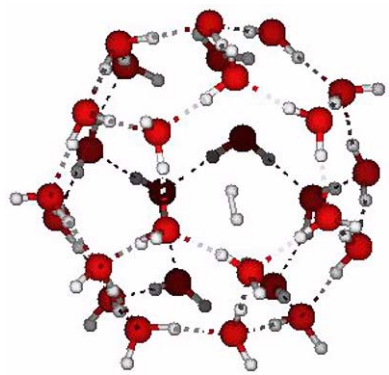
In efforts to utilize hydrogen and methane for energy sources and to understand and predict structure/function relationships for components of cell membranes, a common theme emerges regarding water as the special substance that confers stability to specific structures under specific conditions. For example, a recent study by Mao and Mao [1] shows that water forms clathrate structures around hydrogen molecules at low temperature (T) high pressure (P) and releases hydrogen at high T, lending credibility to the new idea that water could form an environmentally attractive alternative storage compound for hydrogen fuel. We propose to map out the conditions (T, P) for stabilizing hydrate compounds to determine if other guest molecules enhance stability of the molecular water framework and to define the conditions that favor multiple occupancy of the clathrate structures by fuel molecules. We will pursue modeling studies with ab initio methods and experiments by nuclear magnetic resonance to map structures and stabilities of these hydrophobic compounds.

## Accomplishments

We examined, by ab initio methods, the structural and thermodynamic properties of some well characterized systems enclathrated by water to test and develop our ability to predict these properties. First, we considered the clustering reactions of water molecules around several molecular species in liquid water to elucidate the fundamental interactions and clustering statistics of water around small, hydrogen-like molecules. Additionally, we considered water around other small molecules that could be useful as guest additives that might enhance hydrogen occupation. To date, we reproduced experimental water density distribution and hydration free energies for krypton gas and hydroxide ion in water. We additionally examined hydrogen molecule in liquid water, where we know the experimentally determined solubility. Classical studies predict that hydrogen behaves much like krypton, attracting more than a dozen water molecules, while the ab initio studies predict that hydrogen weakly attracts only a handful (four) of water molecules. Studies are in progress to estimate the solubility of hydrogen to determine which model is correct.

We constructed empty type I and II crystal clathrate hydrates and optimized their lattice constants. The predicted constants are similar to experimental values. Although the clathrate hydrates have a more open structure, the predicted binding energies per water molecule are similar to those predicted by Feibelman [2] for ice Ih, the naturally occurring ice crystal.

Finally, we initiated studies to determine the distribution of hydrogen molecule occupations in small and large cages derived from the optimized type II clathrate structure. Current results indicate that only one molecule occupies small cages, while up to four can occupy the larger cages. These findings are consistent with the most recent experimental work, but in marked disagreement with an earlier theoretical work.



*Hydrogen in large clathrate hydrate cage.*

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[2] P. J. Feibelman, “Partial Dissociation of Water on Ru(0001),” *Science*, vol. 295. no. 5552, pp. 99–102, 4 January 2002.

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pation in Clathrate Hydrates," to be published in *Molecular Simulation.*

J. Greathouse, "Atomistic Simulations of Gas Hydrates: Application to Water Desalination," presented at the Mesilla Chemistry Workshop, Las Cruces, NM, April 2005.

S.L. Rempe, J. Clawson, J. Greathouse, D. Sabo, K. Leung, R. Cygan, and T. Alam, "Ab Initio Studies of Gas Stabilities and Occupation in Clathrate Hydrates," presented at the Conference for American Industrial and Chemical Engineers, Cincinnati, OH, October 2005.

S.L. Rempe, "Modeling Real Ions in Complex Environments," presented at the Invited Seminar, PNNL, Richland, WA, May 2005.

# Key Management and Encryption Against Bounded Storage Adversaries

*79805*

*W. E. Anderson, W. D. Neumann, A. J. Lanzone, T. J. Draelos*

## Abstract

Encryption is used in information systems to provide privacy for messages sent between different parties. However, if the secret key is compromised, every message protected by that key could be compromised. To reduce the risk of key exposure, keys are usually updated periodically. Unfortunately, typical rekeying mechanisms may not always be practical or possible.

As cryptographically secure remote sensors become more pervasive for critical infrastructure and homeland security protection, key management for these devices will be a problem. In this project we show how to develop a key management and encryption scheme using a high speed public random source to generate secret keys. Keys derived from a public random source, such as a satellite or deep space radio signal, may be used as one time pads to provide perfect secrecy. This type of scheme removes the burden of typical rekeying mechanisms because devices can rekey themselves. Further, if a device is compromised and its secrets extracted, it is impossible to reconstruct the secret key. By allowing devices to rekey themselves, this scheme will alleviate a significant cost of key management and can have direct application to energy infrastructure security, SCADA (Supervisory Control and Data Acquisition) systems, and sensor or wireless networks.

## Accomplishments

There are several engineering obstacles that need to be solved before key management and encryption under the bounded storage model can be realized. One of the key obstacles hindering its adoption is the construction of a scheme that achieves reliable communication in the event that timing synchronization errors occur. One of the main accomplishments of this project was the development of a new scheme that solves this problem. We show, in general, that message encoding techniques exist under the bounded storage model and provide an arbitrarily small probability of transmission error. We compute the maximum capacity of this channel using the unsynchronized key generator as side channel information at the decoder and provide tight lower bounds for a particular class of key expansion functions that are pseudo-invariant to shift errors. Using our results in combination with the encryption scheme of Dziembowski et al. [1], under the bounded storage model, we can construct a scheme that solves the timing synchronization error problem.

In addition to this work, we conducted a detailed case study of current and future storage technologies. We analyzed the cost, capacity, and storage data rate of various technologies so that precise security parameters can be developed for bounded storage encryption schemes. This will provide an invaluable tool for developing these schemes in practice.

[1] S. Dziembowski and U. Maurer, “Optimal Randomizer Efficiency in the Bounded Storage Model,” *Journal of Cryptology*, vol. 17, no. 1, pp. 5–26, 2004.

# Risk Informed, Decision Making Methodologies for Robust Control of Complex Infrastructures

*79807*

*B. K. Cook, D. G. Robinson, H. E. Link, D. J. Borns, K. L. Stamber, L. R. Phillips, R. A. Laviolette, B. T. Richardson*

## Abstract

Our way of life in the US depends on a complex interdependent system of infrastructures. These infrastructures are currently vulnerable to disruptions that can lead to cascading failures with serious consequences. Our future security and well-being depend upon the successful application of science and technology to develop robust infrastructures that can withstand both terrorist and natural threats. As an integrated human-cyber-physical system, this "living" infrastructure must perceive perturbations, analyze the potential impact of changes to its system health, and respond in a timely manner to minimize the likelihood of realizing some undesirable state or consequence. Active infrastructure control implied in the living infrastructure vision will require the systematic and efficient assessment of infrastructure management options with a risk minimization objective.

The thrust of this project is to develop a representative and holistic modeling framework to assess and manage risk in interdependent infrastructure systems. To develop this capability, we are integrating advancements in complex systems theory, risk management, and distributed sensing and control technologies. The initial research is grounded through the focused study of a specific infrastructure, the bulk power grid. In the first year, we developed a modeling framework by decomposing the power grid into a number of coupled network systems that include the physical grid, a consumer overlay, a control layer, and a market layer. We implemented this abstraction as a dynamic network model that can be used to simulate the progressive failure of the grid and will ultimately allow us to estimate the impact of outages and explore various consequence minimizing management strategies.

Leveraging an improved, systems based understanding of infrastructure behavior and the cost and benefit of mitigating actions, this research will lead to decision making methodologies for infrastructure management that will provide the technical foundation for a new robust infrastructures program at Sandia.

## Accomplishments

Our goal for FY 2005 was to develop the infrastructure risk management framework that will be validated and refined in FY 2006. We made excellent progress toward this goal through the focused study of a specific infrastructure, the bulk power grid, and the abstraction of its relevant structural and dynamic components. We implemented this abstraction as a multi-layer network model that can be used to simulate the progressive failure of the grid. Subsequent extensions of this model will ultimately allow us to estimate the impact of power outages and explore consequence minimizing management strategies.

Key ideas emerged early in our research from an internal infrastructure analysis workshop and literature search of related external research and development projects. Our observations included: (1) limitations of existing risk assessment methodologies when applied to large scale, complex infrastructures, including shortcomings of elicitation approaches and static structural analyses that neglect system dynamics; (2) the necessity of incorporating infrastructure's human, organizational, cyber, and physical components into system models and solutions; (3) the challenge of characterizing threat and the subsequent need to develop solutions robust to varied threat and failure scenarios; (4) business realities that stem from private ownership of the majority of critical infrastructures, and the resulting need for dual benefit solutions that improve both operational reliability and security; (5) varying temporal, spatial, organizational, and other scales that make certain infrastructure systems more amenable to advanced management solutions such as agent-based control; and (6) limited practical engineering applicability of many of the recent complexity science results.

We gave considerable thought to the development of a holistic modeling framework that would capture relevant system features while simplifying analysis. To direct and ground our research, the bulk power grid was selected for focused study. Commonly applied in complex systems analysis, a node-and-link network representation was chosen as the basis for system idealization. Using this approach, we formulated a modeling

framework by decomposing the power grid into a number of coupled network systems that include the physical grid of generation and transmission assets, a consumer overlay dictating demand and spawning consequences from power outages, the human and automated control layer managing the grid, and the market layer representing the buying and selling of bulk power between utilities.

The new National Infrastructure Simulation and Analysis Center's multi-network dynamic modeling platform, Polynet, was selected to implement this grid conceptualization. We successfully integrated an existing alternating current (AC) load flow model into Polynet to simulate the physical grid layer, and implemented a rudimentary control layer to interface with the load flow model. We also extended Polynet with a package that includes an application program interface with a method for loading a power system from an industry standard computational flow dynamics file and a method for running the AC load flow program on the system data. We developed a simulation scheduler that automates successive simulation steps following an initial event (e.g., a user disabling a line through the Polynet graphic user interface) to capture the progressive failure of the power system. We also began an initial characterization of the bulk power market as a preparatory step toward formulating and implementing a simplified but meaningful market model in Polynet.

Risk management will ultimately be realized via the disutility functions being developed jointly with the Massachusetts Institute of Technology. To minimize the aggregate impact of a grid disturbance, knowledge of the consequences associated with the loss of power to a load bus can assist agents in making decisions regarding which components to degrade or trip. This information could also be used to prioritize grid upgrades. We began our initial consequence characterization studies and identified generalized impacts at the load bus level, which will be refined in the coming year.

## Other Communications

D.G. Robinson, "Reliability Analysis of Bulk Power Systems Using Swarm Intelligence," presented at the IEEE RAMS Conference, Washington, DC, January 2005.

H. Link, J. Saia, T. Lane, and R. Laviolette, "Distributed Recommender Systems and the Network Topologies That Love Them," Sandia Report SAND2005-3951, Albuquerque, NM, 2005.

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# Biological Paradigms for Malice Tolerant Computing Infrastructure Assurance

*79808*

*E. E. May, L. G. Pierson, P. J. Robertson*

## Abstract

National and private computational infrastructures are integral in ensuring operation and protection of our energy and critical infrastructures from weapons of mass effects. Malicious attacks on computer systems and associated programs that protect and regulate critical information systems are commonplace. This results in unexpected software behavior and failure of computational systems responsible for securing our water, energy, or nuclear resources. Design and development of computer systems that execute programs reliably in the presence of noise or adversarial attack are important challenges that, if met, will greatly increase the security of our computational infrastructure.

Biological systems exhibit fault tolerance in transmission and execution of their genetic program. Protein production instructions contained in deoxyribonucleic acid (DNA) can be reliably executed after multiple transmissions, surviving natural and malicious mutational errors that occur during an organism's life cycle and over generations of retransmission. We aim to elucidate the underlying architecture of biological communication processes to develop malice tolerant computational infrastructures that perform: (1) real time stream authentication that mirrors biological stream authentication in protein translation processes, (2) redundant instruction mapping similar to genetic code mapping, and (3) instruction control flow analysis analogous to post transcription modification of messenger ribonucleic acid (mRNA). These biological, fault tolerant paradigms have specific information-theoretic coding theory analogues that will be used to design malice tolerant protocols for mass storage and critical computing systems.

## Accomplishments

FY 2005 milestones centered around two project goals: (1) identification of computational analogs to biological malice tolerance and (2) demonstration through simulation of malice tolerance design. We made considerable progress toward achieving both goals.

- Goal 1 Accomplishments

(1) We performed threat analysis for computational infrastructures to scope and guide subsequent work.

(2) We developed a high level description of a malice tolerant system.

(3) We further refined the system description by defining specific procedures to capture and implement malice tolerant characteristics. Procedures include:

(a) DECODE will receive as input a portion of the instruction word and calculate a syndrome (a series of binary bits that indicate the presence of errors in an encoded sequence).

(b) The CORRECT routine will take an instruction word/syndrome pair and attempt to correct the instruction word prior to instruction execution.

(c) Inclusion of a "start" instruction will permit the implementation of a SCAN_FOR_NEXT routine that can be used to find the beginning of the actual program in the event of a threat that changes the size of the instruction.

- Goal 2 Accomplishments

(1) To demonstrate the feasibility of our fault tolerant system, we constructed a software simulation and testing framework for the malice tolerant system.

(2) We prepared the embedded system testbed for hardware demonstrations of malice tolerance concepts ahead of schedule.

(3) We developed a simplified instruction set system for benchmarking and testing in hardware our proposed malice tolerant design.

(4) We developed testing metrics to show a performance comparison of a standard versus proposed malice tolerant system. Testing protocols deal with buffer overflow, code injection, and bit-flip malice scenarios.

# Development of Design and Simulation Models for Large Scale Hydrogen Production Plant Using Nuclear Power

*80568*

*S. B. Rodriguez, R. O. Gauntt, R. K. Cole Jr., L. A. Malczynski*

## Abstract

We are conducting research for the design of a large scale hydrogen production plant and to demonstrate its economic viability and safe operation. The work includes design of the nuclear and chemical component, development of computational models for the nuclear reactor and chemical plant, and the coupling of these models. The simulation model will analyze the three most promising high temperature cycles, namely, the sulfur-iodine (SI) cycle, the calcium bromide/iron oxide cycle, and the Westinghouse cycle. The effects of various reaction temperatures on overall efficiency and hydrogen production will be investigated. Additionally, various ranges of flow rates for both the reaction gases and the heat transfer fluid will be combined with heat exchanger parameters to calculate heat transfer coefficients and assist in designing heat exchangers for the processes in the reactions.

The SI cycle, in particular, has shown high potential for large scale hydrogen production. In order to provide a large power source for the SI cycle, a nuclear plant capable of producing steam up to 850 °C will be considered. The reactor design study will evaluate high temperature gas cooled and molten salt reactor designs and select the optimal design for producing high temperatures in order to maximize hydrogen production on a massive scale. The options in the design include the reactor core design, thermal hydraulics, and safety. Simulation models will be developed for the SI cycle and for the coupled plant analysis. The latter will be based on the MELCOR code and will be coupled to the simulation model for SI plant performance. MELCOR has a strong worldwide reputation as a premiere safety analysis tool for nuclear reactors.

This research will result in the first fully integrated, fully coupled code that can be used to simulate the entire nuclear and thermochemical plant, maximize hydrogen production, and evaluate the potential for safe operation under normal and abnormal conditions. It can also be used to address scalability, hydrogen production cost, and design optimization.

## Accomplishments

(1) We conducted a literature search and critical evaluation of the advances in SI and other cycles for hydrogen production to assess the most promising $H_2$ production cycles. Recent advances related to SI process design, development, and improvements were reviewed. The report highlights technical challenges in thermodynamics, chemical kinetics, side reactions, membrane techniques, and flow-sheet improvement.

(2) We conducted an evaluation of high temperature nuclear reactor system candidates, including neutronic and thermal analysis. Literature on the pebble bed modular reactor (PBMR), the very high temperature gas-cooled reactor (VHTGR), and the high temperature molten-salt reactor (HTMSR) were collected and evaluated. Simple neutronic and thermal analysis for PBMR was conducted.

(3) We completed innovative MELCOR input models for the annular PBMR.

(4) We developed a preliminary SI reaction simulation model for hydrogen production. The simulation model analyzes the three SI reactions at specified lower temperatures and evaluates overall efficiency and hydrogen production. ASPEN PLUS 12.1 was used to simulate simple process systems combined with various process components. SI process simulation for General Atomics' flow sheets was implemented and compared favorably.

(5) We completed a feasibility study on molten-salt-cooled reactor modeling. We completed a literature survey on the properties of various coolants and explored modeling concepts using MELCOR.

## Refereed

S.B. Rodríguez, R.O. Gauntt, S.T. Revankar, and K. Vierow, "Development of Design Simulation Model and Safety Study of Large-Scale Hydrogen Production Using Nuclear Power at Sandia National Laboratories," presented at the New Mexico Hydrogen Business Council, Santa Fe, NM, June 2005.

S.B. Rodríguez, R.O. Gauntt, R.K. Cole, M.A. Modesto, F. Gelbard, L.A. Malczynski, S.T. Revankar,

and K. Vierow, “Development of Design Simulation Model and Safety Study of Large-Scale Hydrogen Production Using Nuclear Power,” presented at the Sandia Cluster Meeting, Albuquerque, NM, June 2005.

S.B. Rodríguez, R.O. Gauntt, S.T. Revankar, and K. Vierow, “MELCOR Modification for Large-Scale Hydrogen Production Using Nuclear Thermochemical Cycles,” to be published in *American Nuclear Society.*

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S.B. Rodríguez, R.O. Gauntt, R. Cole, M. Modesto, F. Gelbard, L. Malczynski, B. Martin, S.T. Revankar, K. Vierow, D. Louie, and L. Archuleta, “Development of Design and Simulation Model and Safety Study of Large-Scale Hydrogen Production Using Nuclear Power,” to be published in *AIChE.*

# Development of a Universal Fuel Processor

*90497*

*J. E. Miller, C. L. Staiger, J. B. Kelley, P. Ho, S. F. Rice, R. A. Kemp, C. J. Cornelius, A. Nanco*

## Abstract

We are developing a new universal liquid fuel and the technology capable of producing this fuel from diverse feedstocks readily available in North America (i.e., petroleum, natural gas, coal, biomass). The market entry point for this universal fuel will be military applications, where the utility of a logistical fuel for all air, ground, naval, and portable power generation has already begun to be recognized. The universal fuel to be developed will have physical properties similar to JP-8 and a distillation profile similar to kerosene or diesel, with a cetane number of roughly 50 and an energy density equivalent to JP-8. This fuel will likely consist primarily of C10-C16 range hydrocarbons, with some branching and ring structures, and relatively little oxygen content. Three critical research areas for the development of this technology were identified in consultation with contacts in the petroleum industry: (1) feedstock transformation, (2) fuel formulation, and (3) fuel characterization.

In the area of feedstock transformation, we are addressing the problem of reactor clogging soot formation during autothermal reforming through a combination of experiments to be performed in pilot scale reactor mock-up and computational modeling. We are also developing a new low cost technology for the separation of oxygen from air that involves a hybrid approach of integrating membrane separations with pressure swing adsorption. In the area of fuel formulation, we are working to advance the synthetic chemistry necessary for the production of a universal fuel via syngas, methanol, and other intermediates that can readily be produced from almost any hydrocarbon feedstock. Fuel specification and characterization will be integrated into these fuel development efforts to ensure that any new chemistries developed ultimately result in a fuel product capable of serving as a logistical fuel.

## Accomplishments

In the area of feedstock transformation, we conducted experiments in an annular laminar flow burner that is representative of industrial equipment for auto thermal reforming (ATR) on methane/oxygen/steam rich flames at pressures up to 500 psi. This higher pressure regime is desirable from an industrial point of view but has been poorly characterized. These experiments aimed to identify the effects of water vapor and elevated pressure on the production of reactor clogging soot. We also began to apply state-of-the-art modeling techniques to the problem of soot formation in ATRs. We demonstrated that oxidant stoichiometry is the dominant parameter influencing soot formation, but also observed that steam can play an important role in minimizing soot. Current models have not captured the role of steam in minimizing soot. Also in the area of feedstock transformation, we started constructing a novel hybrid membrane pressure swing adsorption unit for oxygen recovery from air and have started to synthesize tubular geometry oxygen selective inorganic membranes for evaluation and eventual placement in the unit.

In the area of fuel formulation, we adapted several laboratory reactors for use in this project and used them to evaluate several new chromium based catalysts synthesized in our laboratories. We demonstrated that several of our materials are capable of oligomerizing small molecules and that the zeolitic form of the best materials appears to provide some stability against coking that more open mesoporous structures do not provide. In addition, we demonstrated that framework substitution of the active metal yields a significantly different product distribution than ion-exchanged metals. The framework substituted materials yield oligomeric compounds, while the ion-exchanged materials yield aromatics.

# Rapid Updating of Stochastic Models Using Sensor Information

*90501*

*S. A. McKenna, K. W. Vugrin, E. D. Vugrin*

## Abstract

The goal of this project is to develop procedures for rapidly updating stochastic models of complex phenomena using data collected from sensors in real time, and the implementation of real time control of complex processes encountered in a cross section of earth science and engineering problems. The approach used to achieve this goal is the development of Ensemble Kalman Filters (EnKFs) to both predict and control complex systems in near real time.

Estimation, prediction, and control of many natural and engineered systems are especially difficult due to incomplete knowledge of the system and may be amenable to analysis with EnKFs. The goal of this project is the development of algorithms for real time updating and control of systems using EnKF. If successful, this project will provide a set of tools that will allow for real time updating of models as each new set of observations becomes available and then control of the physical system that is being modeled. This is in contrast to the more commonly employed approach of a one-time optimization of the model parameters after all data have been collected. Initial work has focused on the identification of three test problems and the development of the Kalman filter applications. Initial development of EnKF formulations for each of these three systems has been completed and is discussed below. Current results on the ability of the EnKF to accomplish real time estimation of relatively complex systems includes identifying transport properties of pipe wall material in water distribution systems, conditioning sedimentary process models to observed well logs, and rapid updating of reservoir permeability fields as well test data are obtained. This project is a joint effort between Sandia and the Petroleum and Geosystems Engineering Department at the University of Texas at Austin.

## Accomplishments

We identified three test problems including reconstruction of pipe wall sorption characteristics based on contaminant breakthrough curves in a water distribution system; conditioning of physics based sedimentary depositional models to observed well log data; and conditioning of stochastic permeability fields to observed pressure data.

- *Test Problem 1:* Sorptive transport in pipes. We developed a Kalman filter approach and an EnKF to estimate forward and reverse sorption rates within a pipe containing multiple distinct zones. These estimates are made using continuous concentration data measured by sensors in the pipe at the downstream end of each zone.
- *Test Problem 2:* Conditioning of a sedimentary process model to observed well data. We developed a one-dimensional model for the sedimentary process and EnKF to operate on that model. We demonstrated that EnKF can be used to control parameters in sedimentary process models to create a specified thickness of sedimentary deposit at a certain location in the reservoir. All members of the ensemble converged to true thickness.
- *Test Problem 3:* Updating of reservoir permeability to match observed pressure data. We developed EnKF to work with Eclipse reservoir simulation software. We initiated EnKF updating of the reservoir to match observed pressure data. We are currently in the initial stages of working on a simple reservoir model.

# Analysis of Real-Time Reservoir Monitoring: Reservoirs, Strategies, and Modeling

*90729*

*S. P. Cooper, R. A. Normann, J. C. Lorenz, S. S. Mani, B. G. van Bloemen Waanders, C. J. Weiss*

## Abstract

The goal of this work is to better exploit intelligent oil and gas field information through improved modeling, sensor technology, and process control to increase ultimate recovery from 30% to as much as 65%. We will accelerate the development and use of next generation monitoring technologies such as miniaturized sensors and permanently instrumented wells. These new technologies will be integrated into new simulation paradigms. The capability of simulation to accurately predict recovery depends on improving the quality of input data, and better understanding the impact of uncertainty of the input data, and on refining simulation algorithms.

A primary short term goal of this effort was to better define the required technologies for improved reservoir simulation and modeling. Initial work developed and initiated plans for effective collaborations, and assessed existing tools and algorithms. Value of Information (VOI) decision trees were developed to systematically analyze technologies and their potential to economically solve issues related to recovery. This work is a joint effort between Sandia and the University of Texas at Austin's Bureau of Economic Geology, Department of Petroleum and Geosystems Engineering, and Institute of Computational and Engineering Mathematics.

The impact of successful reservoir monitoring programs and coincident improved reservoir management is the production of additional oil and gas volumes from existing reservoirs, revitalization of nearly depleted reservoirs, possible re-establishment of already abandoned reservoirs, and improved economics for all cases. Smart Well monitoring provides the means to understand how a reservoir process is developing and provides active reservoir management while providing data for developing high fidelity simulation models.

## Accomplishments

Two joint project meetings took place, and work has progressed on the VOI decision tree analyses 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 candidate for industry partnerships and pilot studies using next generation sensors, real time Smart Well technology, and modeling.

Technologies reviewed and evaluated for these potential pilot projects include passive monitoring, improved seismic resolution and four-dimensional seismic, rock physics, joint inversion (which is an integration of seismic, well logs, borehole electromagnetic and interferometric synthetic aperture radar techniques), next generation sensors (enhanced chemical, geophone, seismic, pressure sensors), and permanent sensor placement for real time data retrieval, integration, and modeling. Each of these areas is in the VOI evaluation and planning stage with placement in collaboration with industry and in pilot reservoirs over the next three years.

Analyses to date show our proposed permanently instrumented well technology will improve monitoring. The technology allows the measurement of layer by layer flow, pressure, saturation, and chemical properties in real time. This will dramatically increase the amount of information available, allowing for better resolution, detection of bypass zones, and improved near real time simulation models.

# Merging Spatially Variant Physical Process Models Under an Optimized Systems Dynamics Framework

*90730*

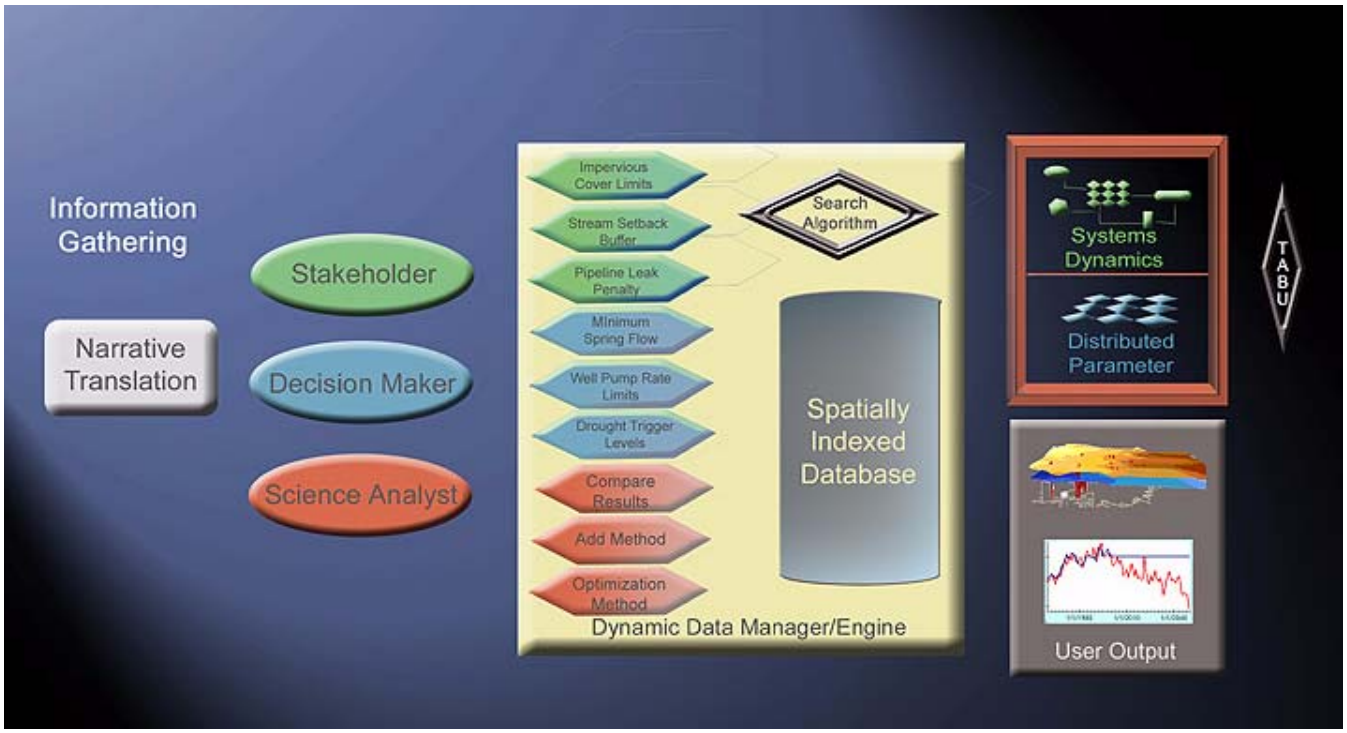
*T. S. Lowry, V. C. Tidwell*


## Abstract

The purpose of this project is to create a prototype integrated modular simulation framework (IMSF) - rapid dispute prevention (RDP) process by integrating spatially detailed water models, systems dynamics models, and stakeholder negotiation processes into one system. The approach creates two "bridges." One bridge spans the gap between data, models, and decision analysis; and the other spans the gap between information, understanding, and decision making. Each bridge enables two-way communication between the connected parts that allows the system to be utilized in an iterative manner, both internally and externally, for model optimization and collaborative negotiations. Several scientific advances were identified for the success of this proof-of-concept effort.

The first advance is 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 second advance is 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 third advance is the development of an intelligence gathering process that scientifically describes and utilizes stakeholder input (data to models). The fourth advance is the design and construction of a decision optimization methodology that promotes the formation of management alternatives based on the latest quantitative hydrogeological data that are amenable to all parties involved (information to understanding to decision making).

This project is a collaborative effort between Sandia and the University of Texas at Austin.


*The IMSF-RDP blueprint showing both the software system architecture and the workflow from information gathering to a negotiation process.*

## Accomplishments

The project focused on the formation of a proof-of-concept project blueprint that addresses two key hurdles. The first hurdle is to understand how we link spatially distributed data and disparate model types into the IMSF. The second hurdle is to understand how we develop stakeholder narratives that can be used to guide model development and usage.

We addressed the first issue through the conceptualization of the IMSF. The IMSF is a software "wrapper" that contains interconnected modules. The modules consist of a data warehouse, spatially distributed numerical models, systems dynamics models, a data exchange manager (DEM), the data exchange controller (DEC), and a graphical user interface (GUI). Together, the DEM and DEC form the communication links between different modules that receive adjustable parameter values from the user, select the appropriate data from the database, format the data in a manner that can be used by the sub-models, run the sub-models, and visualize the results. To date, the IMSF consists of a prototype DEM, a groundwater flow sub-model, a systems dynamics sub-model, the data warehouse, and an operational but simple optimization algorithm.

To address the second issue, we developed a new stakeholder elicitation method (based on a narrative interview process) whereby interested stakeholders are allowed to specify individual objectives, values, and risk tolerances with regard to future resource management. The narrative is used to guide the model development and implementation phase. This process is actively being tested with a group of stakeholders for the Barton Springs Aquifer in Austin, Texas. Toward this end, an initial group meeting was held in early June where the concept of the IMSF-RDP was presented. We gained stakeholder acceptance of the process and agreement to their participation in the project. To date, eight individual elicitations were conducted, and model metrics and components were identified for further development.

## Other Communications

T.S. Lowry, V.C. Tidwell, S. Pierce, J. Sharp, and M. Dulay, "Integration of Spatially Aggregated Physical Process Models Within a Systems Dynamic Framework to Assist the Policy Development and Decision Support Process," presented and published in *Proceedings of the GSA Annual Conference*, Salt Lake City, UT, October 2005.

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# Advanced Spectroscopic Sensors for Proliferation Prevention in Reprocessing Plants

*91664*

*B. B. Cipiti, J. M. Phillips*

## Abstract

Recent interest in reprocessing nuclear fuel in the US has led to advanced separations processes that employ continuous processing and multiple extraction steps. These advanced plants will need to be designed with state-of-the-art instrumentation for materials accountancy and control. This research examined the current and upcoming instrumentation for nuclear materials accountancy for those most suited to the reprocessing environment. Though this topic received attention time and again in the past, new technologies and changing world conditions require a renewed look at this subject. We first identified the needs for the advanced Uranium Extraction Plus (UREX+) separations concept, and completed a literature review of current and upcoming measuring techniques. We compiled a preliminary list of recommended instruments and measurement locations.

## Accomplishments

We investigated a number of different measurement techniques for use in materials accountancy of the advanced UREX+ reprocessing concept. The list expanded significantly once the work started due to the number of measurements taken in current plants. The parameters studied were uncertainty, measurement type, and usefulness in the reprocessing environment.

Current instrumentation reviewed included: level and density indicators, flowmeters, K-Edge densitometry, X-ray fluorescence, hybrid K-Edge, high resolution gamma spectroscopy, isotope dilution gamma spectrometry, mass spectrometry, passive neutron coincidence counting, coulometry, titrimetry, gravimetry, spectrophotometry, atomic emission spectroscopy, calorimetry, chromatography, and alpha monitors.

New instrumentation reviewed included: thermal atomization resonance ionization spectroscopy, nuclear resonance fluorescence, and quantum dots.

We formulated a preliminary list of instrumentation to consider for testing in an advanced plant:

- Spent Nuclear Fuel

High resolution gamma spectrometry, nuclear resonance fluorescence, neutron counting

- Dissolved Fuel

Hybrid K-Edge densitometry, thermal atomization resonance ionization spectroscopy, nuclear resonance fluorescence, inductively coupled plasma mass spectrometry

- Intermediate Processes

Hybrid K-Edge densitometry, isotope dilution gamma spectroscopy, high resolution gamma spectrometry

- U Product

Titrimetry, coulometry, K-Edge densitometry, high resolution gamma spectrometry

- Pu/Np Product

Titrimetry, coulometry, K-Edge densitometry, high resolution gamma spectrometry, neutron coincidence counting

- Cs/Sr Product

High resolution gamma spectrometry

- Am/Cm Product

Hybrid K-Edge densitometry, isotope dilution gamma spectroscopy, alpha spectroscopy

- Waste Streams

High resolution gamma spectrometry, isotope dilution gamma spectroscopy, neutron coincidence counting

- Holdup Measurements

High resolution gamma spectrometry, neutron coincidence counting

# Homeland Security

## Universal Continuous-Flow Bioparticle Processor

*79812*
*E. B. Cummings, D. Reichmuth*

### Abstract

The goal of this project is to create a universal biosample processor system that can process biological samples in complex matrices. This processing includes sample preparation steps like buffer exchange, concentration, particle sorting, labeling and reaction, lysis, separation, and detection. The system has a shared bus architecture through which sample packets are encased as segmented boluses in fluorinated oil (fluorinert) and routed through the bus channel under software control by pressure gradients between devices. The "universality" of the biochemical microprocessor hinges on two factors:

(1) the immiscibility of fluorinert in water and most organic solvents, oils, and biological molecules of clinical and forensic interest, and the absence of surface retention of these samples when they flow past fluorinated surfaces; and

(2) the presence on the sample bus of devices that perform most of the generic processing tasks of practical interest, e.g., mixing, reaction, concentration, storage, detection; and the ready addition of more specific peripherals, e.g., dielectrophoretic spectrometers, antibody assays, enzymatic and catalytic reactors, etc.

All generic functions can be decomposed into comparatively trivial device primitives such as channels, vessels, and heaters, and four new device primitives that must be developed: (1) a voltage controlled valve for the fluorinated oil, (2) a sample segmenter or multiplexer, (3) a sample de-segmenter or demultiplexer, and (4) a packet counting device. Fluidic and electrical interconnections between devices will be made using standard multiple-layer printed circuit board fabrication. The focus in the first year is to demonstrate each primitive, the printed circuit board assembly technique, and the bus architecture validity. In the second and final year, the primitives will be composed into the separate devices of the processor, assembled, and demonstrated in a "hello world" application of the microprocessor: the automated concentration, lysis, labeling, protein extraction, separation, and fluorescence detection of proteomic signatures of bacteria.

### Accomplishments

The project achieved significant progress along three lines:

(1) An innovative redesign of the proposed device architecture that maximizes the impact and marketability of the proposed processor and minimizes risk. Specifically, we

(a) created the conceptual biochemical microprocessor design;

(b) created the detailed schematic designs of each device in the microprocessor; and

(c) distilled the detailed schematics into the lowest number of low risk device primitives that enable all the desired functions: a voltage controlled valve, a sample multiplexer, a sample demultiplexer, and a sample packet counter.

(2) The creation of a multidisciplinary team from industry and academia to accelerate development:

(a) Professors Juan Santiago and Sumita Pennathur, Stanford University

(b) Professors Brian Kirby and Zeke Smith, Cornell University

(c) Professor Kim Williams and post doc, Colorado School of Mines

(d) Professors Jorg Kutter and Detlef Snakenborg, Technical University of Denmark

(e) Sierra Prototype Express, Sunnyvale, CA

(f) Valley Printed Circuitry, Livermore, CA

(3) Progress in demonstration and validation of critical elements:

(a) experimental demonstration of passive multiplexing and demultiplexing of aqueous samples;

(b) demonstration of multiplexing the flow from a separation column and transporting the multiplexed packets along a capillary of about a meter in length to a fluorescence detector, emulating the signal path from a separation column to a detector via the sample bus; and

(c) release of designs for multiplexers and demultiplexer test.

# Security Enabled Programmable Switch for Protection of Distributed Internetworked Computers

*79813*

*J. Van Randwyk, T. J. Toole, L. G. Pierson, P. J. Robertson, B. D. Kucera*

## Abstract

An increasing number of corporate security policies make it desirable to push security closer to the desktop. It is not practical or feasible to place security and monitoring software on all computing devices (e.g., printers, personal digital assistants, copy machines, legacy hardware). We began to prototype a hardware and software architecture that will enforce security policies by pushing security functions closer to the end user, whether in the office or home, without interfering with users' desktop environments. We are developing a specialized programmable Ethernet network switch to achieve this. 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, etc. This ability to remotely update the functionality of the SPS protection device is cryptographically protected from subversion. With this 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, legacy hardware, etc.

## Accomplishments

We began the project by evaluating various security functions for inclusion in the SPS. We determined that an abundance of potential functions exist and that our device should be a platform with the capability to execute these functions rather than a device that has as many functions built in as possible.

We investigated the vendor space for suitable hardware kits on which to build our device. We selected an FPGA (field programmable gate array) kit built by Altera. The kit includes a single Ethernet interface and has the capability to support additional interfaces via a daughter-card feature. We designed and built several interfaces for our development kits and are currently in the testing phase.

We developed an anti-tamper strategy that allows for the ability to securely and remotely reprogram the SPS and leverages previous LDRD work in faithful execution. The code base for the faithful execution work was ported to the newest processor version being used in our development kits.

We selected a DOS (Denial of Service) attack on which to focus our efforts in building a first security function. We have various detection and mitigation techniques for this attack and are going to use a signature based method to control the attack.

We hope to collaborate with an outside partner to continue the SPS software, hardware, and/or architecture development after the term of this research. Recently, we identified a potential collaboration partner, and we are investigating putting code from our project on their hardware and vice-versa.

## Refereed

L.G. Pierson, P.J. Robertson, T.J. Toole, and J.A. Van Randwyk, "Protection of Distributed Internetworked Computers," to be published in *Proceedings of the 2005 IEEE International Carnahan Conference.*

# Rapid Onsite Measurement of Spore Viability

*79814*

*V. A. Vandernoot, T. W. Lane, A. S. Jokerst, S. Branda*

## Abstract

This project addresses problems of consequence mitigation and restoration of facilities following an anthrax attack such as those that occurred since 9/11. Facilities that are contaminated with pathogenic spores, such as *Bacillus anthracis* spores, must be shut down while they are treated with a sporicidal agent and the effectiveness of the treatment is ascertained. This process involves measuring the viability of spore test strips laid out in a grid throughout the facility. The Centers for Disease Control's accepted methodologies require transporting the samples to a laboratory and carrying out a 48 hour outgrowth experiment. This delay greatly extends the amount of time that potentially critical facilities are shut down.

We propose developing a technique that will ultimately lead to a fieldable microfluidic device that can rapidly assess (ideally in less than 30 minutes) spore viability and effectiveness of sporicidal treatment, returning facilities to use in hours, not days. The proposed method will determine the viability of spores by detecting early protein synthesis after chemical germination.

During FY 2005, we established the feasibility of this approach. Proteomic signatures of *Bacillus* spores and vegetative cells were assessed by both slab gel electrophoresis and microchip based gel electrophoresis employing sensitive laser-induced fluorescence detection. We evaluated and optimized the conditions for germination using a number of chemical germinants and ascertained the time course of protein synthesis. We carried out microseparations using both viable spores and spores inactivated by two different methods. A select number of the early synthesis proteins were digested into peptides for analysis by mass spectrometry.

## Accomplishments

- Spore and Vegetative Proteomic Signatures:

We used solubilization conditions compatible with fluorescent labeling and microseparations developed under other funding for this project. Signatures of vegetative cells and spores of *Bacillus anthracis* (delta Sterne), *Bacillus cereus*, and *Bacillus subtilis* were gathered by both slab gel and laser-induced fluorescence gel capillary electrophoresis.

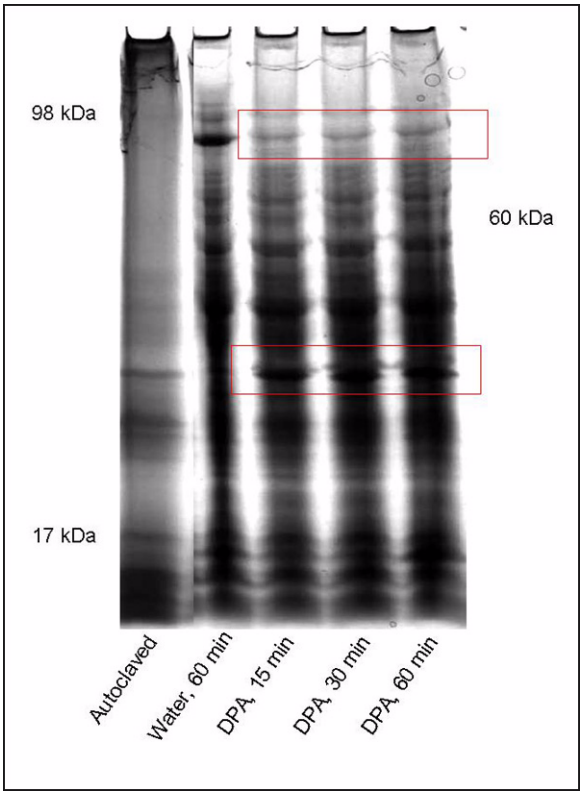
- Develop Rapid Spore Viability:

We evaluated a number of chemical germinants, including alanine for *B. anthracis* [delta Sterne] and *B. subtilis* and inosine for *B. cereus*. First, we heat shocked viable spores to inactivate any vegetative cells that might be contaminating the spores and to help stimulate spores. We incubated the spores with different concentrations of either alanine or inosine and then solubilized and separated them by slab gel electrophoresis. We evaluated time courses ranging from 0 to 120 minutes. We also investigated temperature effects, and, not surprisingly, we found germination at elevated temperatures (37 °C) to be more effective than at room temperature. We evaluated dipicolinic acid, a chemical produced by germinating cells and a known stimulant of *Bacillus* spores. We found it to be much more effective than the discrete amino acid germinants, yielding more distinct new protein synthesis peaks sooner relative to spores in the absence of germinants.

We also carried out control experiments using spores inactivated by both heat (autoclaving) and chemical means (bleach). In both cases, the spores were fully inactivated, as evidenced by a lack of colony formation upon culturing. The signatures did appear somewhat different from viable spores in the case of autoclaving and dramatically different from viable spores in the case of bleached spores. The latter observation is most likely due to a loss of structural integrity resulting from bleaching. In either case, the inactivated spores showed little difference upon addition of the germinant. Viable spores typically showed protein band differences in the 15 minute time frame. This length of incubation time is compatible with the desired goal of a 30 minute viability assessment, given a 5 to 10 minute sample preparation time and a 5 minute microseparation and analysis step.

It is important to note that in all cases the germination was carried out in the absence of culture media. Culture media is undesirable from a proteomic standpoint for two reasons: (1) it can be expected to clutter

the protein signature, requiring that the spores be thoroughly washed prior to analysis; and (2) it represents an additional reagent, one that requires certain storage conditions to maintain efficacy. The presence of culture media would be expected to significantly impact the ultimate utility of this approach.




*SDS-PAGE protein separation of* (a) *viable spores exposed to no germinant for 60 minutes,* (b) *non-viable spores (autoclaved) exposed to dipicolinic acid for 60 minutes, and* (c) *viable spores exposed to dipicolinic acid for 15, 30, and 60 minutes.*

- Mass Spectrometry:

For the purpose of correlating emerging protein bands with known early germination proteins, select bands (a low and a high molecular weight candidate) from the slab gels were digested into peptides, using in-gel digest, for analysis by mass spectrometry. Analysis of the peptide signatures of the specific bands using web based databases is nearing completion.

# Automated Terrorist Threat Detection System

*79815*

*J. J. Carlson, T. H. Ko, J. C. Neely, W. A. Amai*

## Abstract

The purpose of this project is to develop an automated video screening technology to assist security forces in protecting our homeland against terrorist threats. A prevailing threat is 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 required to monitor large areas, the limited number of security personnel employed to protect these areas, and the intense diligence required 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 5 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 that 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, etc.). Detection of background changes and/or unusual foreground activity will be used to alert security forces to the presence and location of potential threats.

## Accomplishments

We made significant technical strides this year. The focus of our efforts was to speed up the background estimation process in the presence of extreme foreground activity. We also examined several techniques to reduce nuisance alarms in our detection algorithms. We initiated 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 the purpose of algorithm development, testing, and evaluation. 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 if the known mixture characteristics in the background monitoring problem could be exploited to reduce the computational requirements. We will be comparing the performance (estimation accuracy, robustness, computational requirements, speed, etc.) of the more traditional methods to those we have developed.

## Other Communications

J.J. Carlson, "Automated Terrorist Threat Detection System," presented at the New Mexico State University Physical Science Laboratory in Las Cruces, NM, April 2005.

# Ion-Induced Gammas for Photofission Interrogation of HEU

*79816*

*A. J. Antolak, D. H. Morse, P. P. Provencio*

## Abstract

High energy photons and neutrons can be used to actively interrogate for heavily shielded special nuclear material (SNM), such as highly enriched uranium (HEU), by detecting prompt and/or delayed induced fission signatures. In this work, we explore a new type of photon source that generates high fluxes of mono-energetic gamma rays from low energy (< 500 keV) proton-induced nuclear reactions. The characteristic energies (4 to 18 MeV) of the gamma rays coincide with the peak of the photonuclear cross section. The source could be designed to produce gamma rays of certain selected energies, thereby improving the probability of detecting shielded HEU or providing a capability to determine enrichment inside sealed containers. We studied the fundamental physics of such an interrogation source through scaled ion accelerator experiments and radiation transport modeling. We used the data to assess gamma and neutron yields, background, and photofission-induced signal levels from several (p,γ) target materials under consideration.

## Accomplishments

We collected gamma spectra from various (p,γ) target materials such as LiF, Teflon, boron carbide, and magnesium. We found that both boron carbide and magnesium have low gamma ray yield that is consistent with the reported $^{11}$B cross section value. We tested magnesium because we could not find a value for its cross section in the literature, but it was reported that a 6.19 MeV gamma ray (in addition to 4.86 MeV and 0.82 MeV gammas) is produced corresponding to the 317 keV resonance of the $^{25}$Mg(p,γ)$^{26}$Al reaction. While our spectra clearly showed the 6.19 MeV gamma ray, we also saw gammas from the higher energy (~8 MeV) branching channels that can occur for the $^{26}$Mg(p,γ)$^{27}$Al reaction. The LiF and Teflon spectra are dominated by the characteristic 6.13 MeV fluorine gamma ray, even observed for 250 keV protons from the accelerator (fluorine has a small resonant cross section of ~0.2 mb at 224 keV proton energy). The resonant reaction for lithium occurs at 441 keV, which accounted for a jump in the measured yield between the 350 keV and 450 keV spectra. The $^{7}$Li reaction is of interest because it produces 17.64 MeV (63% emission per reaction) and 14.74 MeV (37% emission per reaction) gamma rays, which coincide well with the peak of the photofission cross section. We also observed an unidentified, weak low energy nuclear reaction in Teflon that produces ~12 MeV gammas and may be due to a trace impurity in the material.

We conducted experiments to detect prompt neutrons and delayed gammas from induced photofission of depleted uranium ($^{238}$U). 450 keV protons bombarded a LiF target producing 6 (F), 15 (Li), and 18 (Li) MeV gamma rays. A large fraction of these gammas struck a depleted uranium sample that consisted of seven 50 mm diameter x 3 mm thick discs (780 g $^{238}$U) that were stacked outside of the beamline tube 2 cm from the LiF target. A 5 in. x 5 in. NaI gamma detector was positioned 13 cm from the LiF target on the opposite side of the $^{238}$U. The detector was electronically gated with a 35% duty cycle (on 20 microseconds after proton beam off and off 10 microseconds before proton beam on) to capture the short-lived delayed gammas and to avoid any interferences from the source gamma rays. Beneath the gamma source uranium disc experimental arrangement was a plastic moderated $^{3}$He neutron detector array to record the prompt neutron signal. We collected the delayed gamma ray photofission spectra at three proton beam energies (300, 400, and 500 keV) with and without $^{238}$U present. We computed the integrated net signal between 300 keV and 400 keV to be 160 counts/sec, corresponding to the count rate, due primarily to the fluorine reaction at the 340 keV cross section resonance. We planned additional experiments to measure the delayed gamma ray spectra in the 2 to 6 MeV range. For active interrogation, utilizing delayed gamma rays in this energy range has the benefit of producing gamma rays above 3 MeV and, thus, does not interfere with this signature. We found that the cumulative neutron and gamma count rate results were rather low, but the results were nevertheless quite encouraging. For instance, the photonuclear cross sections for both depleted and highly enriched uranium ($^{235}$U) are approximately the same at 6 MeV (p-F reaction), but the $^{235}$U cross section is approximately 2.5 times greater than $^{238}$U at 15 MeV (p-Li reaction). Further,

$^{238}U$ has a significant gamma ray decay background that is almost non-existent for $^{235}U$. Both these effects will improve the signal-to-background ratio for detecting $^{235}U$ signatures.

## Other Communications

A. Antolak, B. Doyle, K.-N. Leung, D. Morse, and P. Provencio, "Active Interrogation Using Low-Energy Nuclear Reactions," in *Proceedings of Penetrating Radiation Systems and Applications VII*, presented at the SPIE Annual Meeting: Optics & Photonics, San Diego, CA, August 2005, CD-ROM.

# Development of Ultraminiaturized Photomultiplier Detectors

*79818*

*M. E. Malinowski, D. M. Skala, C. P. Yang*

## Abstract

Many optical detection technologies require the combination of high gain, low background, and large linear dynamic range signal detection that are only provided by photomultiplier tubes (PMTs). However, many applications are hampered by the relatively large size of commercially available PMTs. In many systems, an ultraminiaturized PMT (a few cubic mm in size) would not only save space, but could enable entirely new system architectures, helping to foster the vision of unobtrusive, ubiquitous, and potentially covert sensors for many applications.

Precise fabrication of the generally complex shapes of dynode structures, in particular, has been one of the major factors limiting miniaturization of PMTs. The geometries of these components are a natural application of LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*) technology, with the shape of the structures and their precise relative locations defined by the high lithographic precision inherent in the LIGA process. We will use advanced LIGA capabilities to design, fabricate, and test this new approach to producing ultraminiaturized photomultiplier detectors.

In FY 2005, we fabricated a prototype ultraminiaturized photomultiplier detector (mPMT) structure and defined a substrate/plating base combination that should yield self-supporting mPMT structures for future LIGA fabrication and subsequent testing.

## Accomplishments

(1) We completed electrostatic modeling of the first mPMT design with a focus lens.

We incorporated focus electrodes into the mPMT design from the start. We also added provisions for mounting a phosphor screen into this design. The original plan called for the inclusion of these design elements in FY 2006.

(2) We designed and made a chrome mask set for mPMT structure and electrode lines. This mask set includes mPMT structures with the same basic "venetian blind" design with three different mPMT component sizes.

(3) We made an x-ray mask for the mPMT structure.

(4) We made x-ray exposures of mPMT in PMMA aligned to base electrode lines and developed PMMA molds.

We tried two different aligned exposure techniques: a mechanical, pin-aligner technique and an optical technique using a commercially available mask aligner. The optical technique produced the best alignment and is currently used for alignments.

(5) We chose a substrate/plating base combination.

The mPMT structure requires not only that the structure be plated in a geometry aligned atop the electrode lines, but also that the structure adhere strongly to the plating base/substrate. We tried two different insulating substrate materials (quartz and alumina) as substrates and tried two different plating bases (Ti/Cu/Ti and $TiO_x$/Ti/Au/Cr) as candidate systems. We found that the quartz substrates cracked during plating of the structures; this failure removed quartz from further consideration. In addition, using a Ti/Cu/Ti plating base on alumina was unacceptable because of base failures caused by the non-conformal coating of the plating base on the alumina. These results suggested that the best combination would be the $TiO_x$/Ti/Au/Cr plating base atop an alumina substrate.

(6) We successfully fabricated a prototype mPMT structure on a silicon substrate.

We successfully demonstrated plating of the mPMT structure using $TiO_x$/Ti/Au/Cr on a silicon substrate and made mPMT arrays approximately 500 microns high. These structures showed that the thick $TiO_x$/Ti/Au/Cr plating base, with about 6 microns of $TiO_x$+Ti, could be used as a stable, patterned plating base. Moreover, these same structures also survived wafer dicing, resulting in discrete mPMT structure chips about 12 mm square. This work also showed that the focus electrode elements in the original mPMT design structure were too fragile and could be bent in handling. The next mPMT mask design will incorporate larger diameter focus elements.

(7) We designed, built, and tested a system for measuring secondary electron yields (SEYs) of dynode coating materials. MgO will be the first SEY coating

tested. It can be selectively deposited onto the dynodes by an electrodeposition process. We also created a deposition cell.

Using the results from (5) and (6) above, we made the first attempt at forming the mPMT atop the patterned $TiO_x$/Ti/Au/Cr plating base on an alumina substrate. Although alignment, exposure, and plating in nickel were successful, the mPMT structure separated from the thin Cu strike applied atop the $TiO_x$ before plating. The exact cause for this separation is not known, but it is likely that the Ti in the plating base layer was not sufficiently oxidized, leading to an adhesion failure. However, results on the silicon wafer showed that the TiOx/Ti/Au/Cr plating base will work, given a properly formed $TiO_x$ layer. We will try mPMT fabrications on the TiOx/Ti/Au/Cr plating base atop alumina again, with more attention given to the Ti oxidation. This fabrication procedure should soon yield a complete mPMT structure atop an insulating alumina substrate.

# Software Architecture for Scalable and Adaptive State Management in Support of Decision Analysis Simulations

*79819*

*S. D. Kleban, A. S. Yoshimura, M. S. Bastian, M. E. Goldsby, H. R. Ammerlahn, T. J. Sa, D. D. Djordjevich, J. M. Linebarger, M. F. Hawley, P. C. Moore, D. Fellig*

## Abstract

The purpose of this project was to further develop and demonstrate integrating software architecture frameworks for distributed simulation and distributed collaboration. The primary objective was a demonstration of both a federation of distributed simulations (BioDAC and a proxy for the N-ABLE™ simulation engine) and a federation of distributed collaborative simulation analysis communities in the context of the same integrated scenario, which was the release of smallpox in San Diego, California. To our knowledge, this was the first time such a combination of federations under a single scenario was ever demonstrated. A secondary objective was the creation of the standalone GroupMeld™ collaboration client, which uses the GroupMeld synchronous collaboration framework. In addition, a small pilot experiment that used both integrating frameworks allowed a greater range of crisis management options to be performed and evaluated than would have been possible without the use of the frameworks.

## Accomplishments

The primary research achievement of the project was the creation of two federations linked together by the unifying scenario: a federation of distributed simulations and a federation of distributed simulation analysis communities. We created the federation of two distributed simulations to generate and respond to the scenario. The first simulation was BioDAC, a crisis management simulation for biological weapons of mass destruction, which ran in Sandia California. The second was N-ABLE (more precisely, a proxy for N-ABLE), an agent based microeconomic simulation, which ran in Sandia New Mexico. The IDSim distributed simulation framework was used to create a unified federation of these two simulations. The two simulation analysis communities were those associated with the BioDAC and N-ABLE simulations, respectively. We used the GroupMeld synchronous collaboration framework to create a collaborative federation of these two simulation analysis communities. To our knowledge, this is the first time that these two kinds of federations linked together in a common scenario were ever demonstrated.

Another significant accomplishment of the project was the creation of a standalone GroupMeld client application. This client application uses the GroupMeld collaboration framework to provide a rich set of collaboration services and is available on demand from an internal Sandia Web site.

The final accomplishment was a small pilot experiment that used both integrating frameworks. This demonstrated that the use of the integrating architectures allowed a greater range of crisis management options to be performed and evaluated than would have been possible without the use of the frameworks.

# Portable Medical Diagnostic System for Detection of Presymptomatic Biomarkers of Chem/Bioagent Exposure

*79820*

*A. K. Singh, G. J. Fiechtner, B. A. Simmons, A. E. Herr, L. M. Barrett*

## Abstract

We propose to develop a portable device for rapid screening and triaging of a population after a suspected incident of bioterrorism. The device will carry out multi-analyte detection rapidly (in minutes) using a tiny amount of blood or saliva. The presymptomatic detection will be based on detection of early response immune biomarkers such as cytokines. 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, toxins, and 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 will be analyzed using microarrays of immobilized receptors.

## Accomplishments

- Analysis of Blood in Microchips:

The first step in developing a chip based diagnostic was to test our chips and other methods for compatibility with blood and other bodily fluids such as saliva. Although our group has years of experience with microchips for analysis of environmental samples such as water and aerosol eluent, our chips and methods had not been developed or tested for more complex samples such as blood. Blood is arguably the most complex matrix, as it contains over 10,000 constituents that span the size range from multiple microns (red and white blood cells) to a few nanometers (ions), and molecules that are very hydrophobic (lipids) to amphiphilic (proteins) to hydrophilic (salt ions). It also contains molecules that are present in micromolar to millimolar range (salts, albumin) and proteins that are present in picomolar concentrations, such as cytokines. We addressed the problem of Joule heating by developing assays that are completed at low field strengths. We also developed surface coatings and castable polymers (polyacrylamide) that are hydrophilic and do not show appreciable adsorption. The assays are also performed in buffers and pH that do not disrupt activity of analytes.

*Researcher Anup Singh inserts an immunoassay chip into the microfluidic module of the hand-held device.*

- Development of a Miniaturized Fluorescence Detector for Red Wavelengths:

The traditional µChemLab™ detector uses a diode laser with excitation wavelength at 405 nm. In preliminary experiments with animal blood serum, we labeled the proteins in serum with fluorescamine and performed a gel electrophoretic separation of serum proteins. We observed that the background fluorescence was high compared to that with buffer. Blood has many components, such as bilirubin, that fluoresce when excited by ultraviolet and low visible wavelengths. This led to a decrease in detection sensitivity at 405 nm excitation. Biological molecules have been reported to have lower fluorescence in the red spectrum. Hence, we used a HeNe laser to perform detection at 633 nm

and observed much lower background for detection of Alexa-647 labeled ovalbumin in spiked bovine serum. Low nM protein could be easily detected. Consequently, to meet the goals of this and other projects that use bodily fluids as samples, we developed a miniaturized red-diode based detector. This detector enabled detection sensitivity of approximately 10 nM Alexa-647 labeled proteins, which is approximately tenfold higher than sensitivity obtained with a bench-top HeNe laser setup.

- Microchip Based Immunoassays for Presymptomatic Markers:

We developed sensitive immunoassays for a number of analytes that could be potential biomarkers of exposure to a bioagent. It should be noted that at this point none of these biomarkers have been correlated with exposure to any specific agent. Our goal was to demonstrate proof-of-principle with potential biomarkers. We selected TNF and IL-6 as representative analytes, as many researchers have shown that in animal studies exposure to anthrax toxin leads to elevated levels of these cytokines. We completed these immunoassays in minutes, compared to hours required for conventional enzyme linked immunosorbent assays. Limits of detection were 4 nM for IL-6 and 700 pM for TNF. We also developed immunoassays for toxins such as tetanus (surrogate for botulinum) and anthrax toxin protective antigen.

# Exploratory Research into Pathogen Surface Interactions

*87787*

*T. W. Lane, S. J. Altman, R. Rebeil*


## Abstract

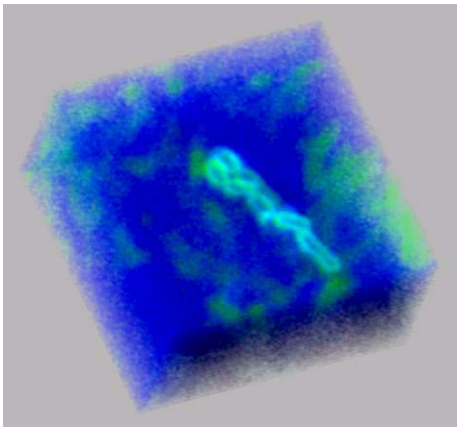
The goal of this research was to characterize bacterial cell surface interactions with other cells and material surfaces to assist in detection and mitigation strategies for pathogens. At the molecular level, the primary goal was to understand how the important pathogens *Francisella tularensis* and *Legionella pneumophila* interact with their environment in biofilms, protozoa, and macrophage. Ultimately, the results will help discriminate between virulent and nonvirulent states of the pathogen.

The research team achieved growth of both drinking water biofilms and monospecific biofilms of *Legionella pneumophila*. We carried out preliminary comparative proteomic analyses on planktonic and biofilm-associated *Legionella*. We obtained the Utah 112 strain of *Francisella novicida* and carried out preliminary culture and comparative proteomic analyses. We conducted comprehensive literature searches and data mining on all research topics.


## Accomplishments

We successfully grew biofilms from drinking water. We first dechlorinated the water by running it through two columns of granulated activated carbon. We then mixed the drinking water with an equal volume of 1:100 Trypticase Soy Broth solution and dripped it into a drip-flow reactor. With this higher concentration of nutrient solution, we were able to grow very thick biofilms. We used hyperspectral imaging with multivariate analysis techniques (multivariate curve resolution) to image the drinking water biofilms. Hyperspectral imaging produced excellent images of the bacteria in the drinking water biofilms. There is also evidence from the hyperspectral imaging that some substance (perhaps extracellular matrix) is integrated with the outer parts of some of the microorganisms.

We cultured *L. pneumophila* from frozen stocks in buffered yeast extract liquid media supplemented with activated charcoal, L-cysteine, and ferric pyrophosphate. Cells grown in tubes without agitation formed a robust and highly cohesive biofilm with few planktonic cells present in the media. To stimulate planktonic cell formation, we placed culture tubes in shaker incubators (37 ºC, 250 rpm) to simulate turbulent flow. We extracted total proteins from cells and analyzed them by two-dimensional electrophoresis.



*Hyperspectral image of extra-cellular matrix (blue) and microorganisms (green) in a drinking water biofilm. The cyan shows the overlap between the green and blue.*

*L. pneumophila* grew in large cohesive macrocolonies. To confirm that the macrocolony observed was a biofilm, Congo Red dye was added to the media. (Congo Red has been shown to stain bacterial colonies of organisms generating biofilms.) The *L. pneumophila* macrocolonies bound most of the Congo Red, which binds to the extrapolysaccharide matrix of biofilms. To confirm our findings, we placed fragments of the Congo Red stained macrocolony on a slide and examined them via light and fluorescent microscopy. As expected, bacterial cells were suspended and held together by an amorphous matrix, presumably an extracellular polysaccharide, that fluoresced due to the presence of the Congo Red dye. We extracted proteins from these macrocolonies for further study.

We placed tubes that had just been inoculated with *L. pneumophila* frozen stocks in an incubator/shaker (250 rpm) to simulate growth under turbulent flow. The *L. pneumophila* grown under turbulent flow yielded a turbid culture that did not contain macrocolonies. In addition, very little Congo Red dye bound to the cells, indicating that the cells were indeed in a planktonic

state. Further optimization will be required, yet clearly planktonic and biofilm cells of *L. pneumophila* display different protein contents even though they are grown in identical nutrient media.

We completed a comprehensive literature search for both the biofilms and the *Francisella tularensis* efforts of this project. We obtained permits from the Department of Agriculture and the Centers for Disease Control for the transfer of *F. tularensis* subspecies novicida strain Utah 112 (*F. novicida)* to Sandia and registered with the National Institutes of Health Biodefense and Emerging Infections Research Resources Repository. After we met all of these regulatory requirements, the strain was transferred to Sandia, and we initiated cell culture experiments. We grew *F. novicida* under a variety of culture conditions and harvested it at different growth phases. We performed proteomic analysis of cells from each sample by gel electrophoresis.

# Military Technologies and Applications

## Compact Terahertz (THz) Sources for Emerging Threats Applications

*52582*

*A. Mar, G. A. Vawter, J. R. Wendt, F. J. Zutavern, M. W. O'Malley*

### Abstract

The purpose of this project is to develop options for compact sources of electromagnetic (EM) radiation in the "THz gap" of the EM spectrum for covert communications and directed energy. This spectrum is very attractive for covert applications due to the lack of readily available instrumentation and the potential compactness of highly directional transmitters. Secure communications at THz frequencies could employ strong absorption lines in atmospheric propagation to confine the range of potential interception, as required, for example, by Future Combat Systems. The highly directional nature of THz radiation could also enhance disruption effects on radio frequency (RF) communications (a concept we demonstrated at microwave frequencies) and other RF electronics. Compact and relatively inexpensive sources will also be required for large scale deployment of THz based technologies, such as imaging devices for homeland defense.

Currently, THz research typically involves the use of large and costly laboratory femtosecond laser systems. We will develop heterodyned continuous wave (CW) lasers and harmonic mode-locking of diode lasers to generate THz with compact systems of turnkey simplicity. We will combine such new compact optical sources with photoconductive semiconductor switch (PCSS) technology for THz generation and detection.

THz technology has direct implications for many applications involving miniaturized high voltage electronics, such as neutron generators, radars, high power microwave (HPM) systems, and lasers. Imaging based on THz radiation (T-rays) finds use in the inspection and qualification of weapon components such as neutron tubes and firing sets. T-rays penetrate through nonconducting materials, such as those used in high voltage potting and image materials, by the variation of the complex dielectric constant. T-ray images are thus complementary to x-rays, whose image contrast is provided by the atomic number of the materials imaged.

### Accomplishments

We assessed power output limitations and scaling limits of a multi-THz system. We fabricated and characterized a third generation of devices for the highest possible output power and operating frequency. We developed an etching process using new chemically-assisted ion beam etching (CAIBE). The rib waveguide etch is performing well, and the first generation lasers work. The intracavity reflector (ICR) etch looks smooth and vertical, and work is ongoing to further deepen slots-devices.

We began development of first generation masks without ICRs. We conducted optical measurements of colliding pulse mode-locked (CPM) THz laser operation and found that colliding pulse mode-locking does not depend on ICR structure. We observed mode-beating/locking at 267 GHz in optical spectra using a saturable absorber. The ultrasensitive scanning autocorrelator is operational.

Toward the development of the lock-in detection, two-photon absorption (TPA) detector, we accomplished the following:

- We measured 20 fJ pulse energy from 30 ps pulses.
- We made a preliminary measurement of 0.267 THz using the CPM device.
- We began characterizing neutron bombarded GaAs carrier lifetimes using femtosecond laser-based pump-probe reflectometers.

We also developed critical tools for optical testing of these devices, including an ultrahigh sensitivity scanning autocorrelator. We are testing CPM devices at the 2nd harmonic (~280 GHz).

# Improving Human/System Interactions in Systems-of-Systems

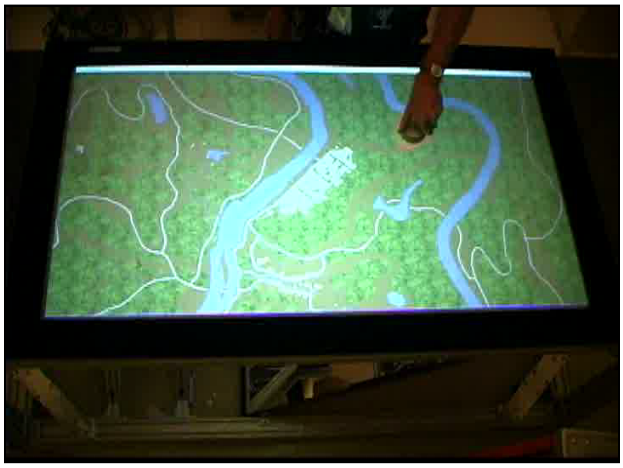
*52585*

*S. Tucker, D. P. Miller, M. J. McDonald, B. E. Hart, M. J. Gardner, M. L. Bernard, S. P. Holman, B. F. Clark, T. Smith, K. Summers*

## Abstract

A number of applications are limited primarily by their human-system interfaces (HSIs). Unlike machines, humans are adept at conveying information between themselves using a variety of techniques, including speech, gesture, using props (tangibles), and drawing. By coupling technologies that mimic these interactions, we are developing interface technologies for complex systems that are both intuitive and user-centric in design. Intuitive interfaces become more appealing with increasing system complexity, such as with large system-of-systems (SoS) tools. While this work is useful to a variety of human-system interactions, our focus will continue to be on the development of an HSI for modeling and simulation (M&S), especially analytic tools. To date, the capability of most M&S applications is underutilized due to lack of tools that enable effective management by humans. The push to utilize M&S for complex analytics has resulted in tools that are usable only by specially trained modelers. This paradigm leaves the end-user of the analyses, the person posing the questions, needing assistance from modelers to have their questions answered. The main premise of this project is to allow a decision maker who has little familiarity with M&S to be able to interact with the simulation in a naturalistic manner, similar to ways humans communicate about ideas to others. To meet this goal, we developed a user-centric HSI prototype based on the taxonomy of observed human interactions integrated with a three-dimensional (3-D) tangible interface coupled with voice and gesture recognition. For the M&S framework, we used Sandia's Umbra computer code and added object oriented containers to support the easy creation of hierarchical systems representations. We are implementing the concepts of architectural description languages (ADLs) utilizing XML, based on Carnegie Mellon University's ACME code. Basic gestures are implicit in the functional design of the tangibles. The current voice command implementation is based on a commercial voice menuing product, Lumenvox™.

*Users interacting with the tangible table.*



*Movie of interaction with the tangible table.*

## Accomplishments

We developed our next generation tangible interface based on hardware tracking of tangible "pucks" on a bottom projection table that interacts with Sandia's Umbra environment. For the pucks, we are using a Phasespace™ system that uses light emitting diodes and sets of crossed linear visible sensors. Much of the effort was to implement the pucks as a wireless radio frequency (RF) system. The RF system allows 40 pucks to be tracked in 6 degrees of freedom. These pucks are tracked at 480 Hz and to submillimeter accuracy. This capability allows for the interpretation of subtle hand gestures. Gestures include rotation, lifting the puck off the surface (contact switch), and separate

pressure sensitive switches on top of the puck as well as around the circumference. The tangible table is waist high for an average standing person, with a 30 x 55 inch high definition television projector displayed from below. Additionally, a projector above projects information onto the puck about its current state, i.e., what simulation entity a puck represents. A commercial voice menuing system, Lumenvox, is used to communicate the user's desired interaction with the puck and simulation. We developed an Umbra graphical user interface extension that allows user interaction with Umbra simulation objects and supports high level architecture (HLA) interface with remote Umbra simulations. All internal systems communication between servers on the tangible table is via HLA. This interaction allows any arbitrary puck to be associated with an Umbra object (model component). It uses the puck to modify physical characteristics of the object by performing gestures with the puck. In use, a context specific puck menu displayed on a separate video screen allows the user to select from several available actions or puck functions. This allows small groups to collaborate and manipulate the simulation by manipulating the pucks around the display table.

We are working with the University of Southern California's (USC's) Integrated Media Sciences Center (IMSC). IMSC has advanced voice and gesture recognition technologies. Visually recognizing the emotional state of a user in an interactive environment is of critical importance for developing systems that are aware of the user's state and cognitive processing capabilities. Human posture and gestures are important components of human-to-human communication and are good indicators of the user's state. Gestures are commonly used to point to specific locations, attract other people's attention, or to indicate agreement or disagreement. USC can provide methods to automatically extract and understand the nonverbal communication cues from a set of cameras observing the user in a simulation room. Also, their voice recognition work focuses on enabling natural and robust spoken language interactions by itself and in combination with other sensory and non-sensory input modalities (such as gesture) for human-machine communication. IMSC will provide a research plan for future work to integrate their gesture and voice interfaces with the tangible table.

Additionally, we are making modifications to Umbra, including implementation of XML parsers and our implementation of an ADL to better address issues of representing entities, behaviors, and connections in more complex hierarchical system-of-systems models. The design of our system representation closely follows design concepts borrowed from Carnegie Mellon University's ADL called ACME, developed for modeling complex architectures.

We based our FY 2005 demonstration on an urban terrain scenario: the user was able to interact with an unmanned aerial vehicle model, with visual sensors and communications repeaters, and multiple ground based sensors to interactively track an adversary vehicle in real time. While friendly vehicles were visible at all times, with force tracking technologies, the adversary was visible only when the user properly configured the available sensors and communication technologies. This allows a user to discover complex system interactions in an intuitive lifelike manner.

# Secure Chaotic Communications

*52587*

*R. A. Salazar, L. L. Molina, L. D. Bacon*

## Abstract

The goal of this project is to develop a secure chaos-modulation based communications system with applications to nuclear weapons, military systems such as Future Combat Systems, and commercial systems requiring a high level of security. We devoted the first year of the project to studying existing chaotic schemes and their suitability for use in communications applications. Based on this study, we selected a chaotic frequency-hopping scheme as the basis for a secure communication system. During the second year of this study, we designed a two-way communication system based upon frequency hopping.

We based our communication system design on a chaotic cryptographic scheme using the logistic equation. The flexibility of our design allows us to use other equations. Our approach is a private-key scheme since it uses the same key for both encryption and decryption. The key is composed of the initial conditions and system parameters used in the logistic equation. We based our design on an algorithm that is considerably faster than algorithms proposed by others. This algorithm will improve data rates and produce higher entropy.

During our third year, we will fully characterize and analyze the performance of our design. We will conduct simulations to evaluate the performance of the system in terms of both standard communication performance and cryptanalysis. Standard communication performance includes: spectral efficiency and bit error rate (BER) as a function of bit energy (Eb). For cryptanalysis performance, we will evaluate the difficulty of determining the coding key that is required to decrypt ciphertext.

## Accomplishments

In FY 2005, we achieved system security by implementing a spread-spectrum technique called frequency hopping. Frequency hopping is popular because it has good propagation properties. However, not all frequency hopping systems are secure. From a security viewpoint, if one is able to follow the frequency hopping pattern, then one can use the pattern to anticipate future transmissions. Given that one can anticipate future transmissions, one can capture enough of each transmission to determine the entire message. In our application, the sequence of hopping frequencies follows a chaotic pattern that is aperiodic; hence, the sequence never repeats and the message is never compromised.

This implementation uses a private-key scheme, meaning that the same key encrypts and decrypts the coded message. The key is composed of the initial conditions and system parameters used in a chaotic equation. The message transmitted contains information needed to generate the plaintext. The algorithm we implemented allows high data rates and higher entropy than previously obtained. The BER performance of this scheme is much better than other chaotic schemes. In fact, the BER rate is the same as a normal frequency shift keying (FSK) modulated system. The system we built and tested uses FSK as part of its modulation. This spread-spectrum technique has the added benefit of low probability intercept.

# Microflame-Based Detector Suite for Universal Gas Sensing

*52590*

*R. P. Manginell, P. G. Clem, M. W. Moorman, D. R. Wheeler, M. Okandan, S. K. Showalter, P. R. Lewis*

## Abstract

We developed a microflame-based detector suite for sensing a broad range of chemical analytes that combines calorimetry, flame ionization detection (FID), nitrogen-phosphorous detection (NPD), and flame photometric detection (FPD) modes into a platform based on a microcombustor. The microcombustor consists of a micromachined microhotplate with a catalyst or low work function material added to its surface. In the NPD mode, a low work function material selectively ionizes chemical analytes. For all other modes, we use a supported catalyst such as platinum/alumina. The microcombustor design permits rapid, efficient heating of the deposited film at low power. To perform calorimetric detection of analytes, we measure the change in power required to maintain the resistive microhotplate heater at a constant temperature. For FID and NPD modes, electrodes are placed around the microcombustor flame zone, and an electrometer circuit measures the production of ions. For FPD, the flame zone is optically interrogated to search for light emission indicative of de-excitation of flame produced analyte compounds. The calorimetric and FID modes respond generally to all hydrocarbons, while sulfur compounds only alarm in the calorimetric mode, providing speciation. The NPD mode provides 10,000:1 selectivity of nitrogen and phosphorous compounds over hydrocarbons. The FPD can distinguish between sulfur and phosphorous compounds. Importantly, we can establish all detection modes on one convenient microcombustor platform. In fact, we can achieve the calorimetric, FID, and FPD modes simultaneously on only one microcombustor. Therefore, it is possible to make a very universal chemical detector array with as little as two microcombustor elements. We completed a demonstration of the performance of the microcombustor in each of the detection modes.

## Accomplishments

By placing electrodes and optical detectors around the flame zone of a microcombustor, we constructed and tested a microFID, microNPD, and microFPD. With a minimum of two such microcombustors, one with a catalyst to generate a flame and one with a low work function material to generate an NPD, we can perform simultaneous FID, NPD, FPD, and calorimetry. The addition of another inactive microcombustor would allow cancellation of common-mode effects such as ambient temperature and flow variations. Thus, a two or three element array of microcombustors would provide a wide variety of response from general hydrocarbon detection to specific detection of N, P, and S compounds. This is important since we can obtain high selectivity with a device having a footprint about the size of a quarter, including packaging. Low hydrogen use and power consumption of this device make it amenable to portable use, and the selectivity of the array will allow it to perform well in the real world. This is especially true if combined with a micro gas chromatograph to perform field separations. The various modes of detection of this platform will allow application to several important areas, including homeland security, sulfur detection, and petrochemicals.

## Refereed

R. Manginell, C. Washburn, M. Moorman, G. Shelmadine, and T. Hamilton, “MicroFID Using a Microcombustor,” to be published in *Proceedings of the IEEE 2005 Sensors Conference.*

# Nonlethal Technologies for the War on Terrorism

*62827*

*P. D. Coleman, M. L. Weyn, T. Lemp, D. B. Seidel, M. C. Wanke, V. M. Hietala*

## Abstract

This project will establish the technical feasibility for a dramatically new method to counter elements of the terrorist threat and to affect the way we engage adversaries when lethal force is not appropriate. A nonlethal response opens a range of options for dealing with the situation by temporarily incapacitating hostiles without injury. Short wavelength radio frequency (RF) has been shown to create a strong flee reaction. However, present systems designed for this RF application are based on vacuum tube technology and are expensive, large, and fragile. Further, conventional semiconductors do not have the required power. This project proposes to build a microelectromechanical systems (MEMS) fabricated micro vacuum electronic source that could be arrayed to yield a robust, high power, and relatively inexpensive alternative to present systems. These sources can find uses in access denial (military and civilian use), higher resolution synthetic aperture radar (SAR), wideband communications, and imaging through obscurants (including clothing and epoxies used in weapons manufacturing).

The main Department of Energy use would be in the protection of weapons and special nuclear materials that would allow for a cost effective solution to ever increasing security costs. In the Department of Defense, the main use would be in nonlethal weapons.

## Accomplishments

We accomplished work in two areas in FY2005: simulations of an advanced split cavity oscillator (SCO) design, and MEMS fabrication of the baseline transit time oscillator (TTO) design.

Previous work indicated that, for typical beam parameters available from field emission arrays (FEAs) and for the simple baseline cavity geometry, the TTO instability was weak. As a result, overcoming wall losses could be problematic.

We investigated an SCO cavity design as a second generation device that could possibly provide a more robust instability. We performed extensive particle-in-cell simulations to optimize the cavity geometry. We calculated instability growth rates for various beam currents and beam radii. Although oscillations were well defined, excessive currents (beyond the capabilities of the FEA) were required to overcome wall losses.

The fabrication of the baseline device can be conveniently separated into four processes: (1) the substrate with sealed electrically conductive vias; (2) the FEA fabrication and characterization; (3) the lower wall fabrication, including the pocket that locates and stabilizes the FEA prior to brazing it in place; and (4) the top fabrication, including the top electrical FEA contact, diffusion bonding locating ring, and antenna ports. We successfully built the substrate and completed the FEA fabrication and top fabrication. However, the lower wall fabrication proved to be difficult. The lower wall was to be 750 µm high with a 200 µm wall thickness. We found the structure to be too narrow to be effectively processed as planned (a series of 200 micron high negative resist definitions). Residual resist and lapping residue was problematic. We identified alternative approaches that look like good solutions for building the device.

# Advancements in Sensing and Perception Using Structured Lighting Techniques and an Innovative Design Tool

*67029*

*D. D. Padilla, P. A. Davidson Jr., D. K. Novick, C. W. Wilson, J. J. Carlson*

## Abstract

There is an ever increasing need for robust, autonomous ground vehicles for counter terrorism and defense missions. After nearly 30 years of government sponsored research, it is undisputed that significant advancements in sensing and perception are still necessary. We developed an innovative, advanced sensing technology for national security missions. The principal goal of this project was to develop an eye-safe, robust, low cost, lightweight, three-dimensional (3-D) structured lighting sensor for use in broad daylight outdoor applications. Currently available laser scanners are slow, bulky, heavy, expensive, fragile, short-range, sensitive to vibration (highly problematic for moving platforms), and unreliable for outdoor use in bright sunlight conditions. Eye safety issues are a primary concern for currently available laser based sensors. Passive stereo imaging sensors are available for 3-D sensing but suffer from limitations; they are computationally intensive, they require a lighted environment (natural or man-made light source), and they do not work for many scenes or regions lacking texture or with ambiguous texture. Our approach leverages the advanced capabilities of modern charge-coupled device (CCD) camera technology and expertise in 3-D world modeling, mapping, and analysis using structured lighting. This project will extend the lifecycle of our current technology and open a new capability for outdoor 3-D mapping. Potential applications include precision mapping, autonomous navigation, dexterous manipulation, surveillance and reconnaissance, part inspection, geometric modeling, laser based 3-D volumetric imaging, simultaneous localization and mapping (SLAM), aiding first responders, and supporting soldiers with helmet-mounted laser radar (ladar) for 3-D mapping in urban environment scenarios. The technology developed in this project overcomes the limitations of current laser based 3-D sensors and contributes to the realization of intelligent machine systems reducing manpower need.

## Accomplishments

We implemented a polarization sensitive system to increase the signal-to-clutter ratio. The kinds of surfaces we imaged were natural and man-made, such as asphalt, concrete, dirt, and grass. We made a series of measurements for each of the materials studied to find the best configuration for the polarizing system and also to find out the potential improvement in the signal-to-clutter ratio. We divided this process into three parts: characterization of the reflected sunlight, characterization of the reflected laser light, and measurement of the improvement in the signal-to-clutter ratio.

We also implemented a synchronized laser pulse-width modulation technique. To employ this method, it was necessary to synchronize the laser pulsing with the exposure period of the camera. We used an LM1881 Video Sync Separator to determine when the CCD sensor of the camera was exposed (i.e., when the shutter was open) during the odd and even fields of each frame. The LM1881 extracts the video synchronization signal from a standard NTSC (National Television Systems Committee/RS-170A color standard) camera, allowing us to determine how long the camera shutter is open after the vertical retrace pulse. We determined the timing by pointing the camera directly at a light emitting diode (LED) that was being pulsed by the MSP430 Texas Instruments microcontroller. The exposure time of the camera was set to 1/250 of a second, and the LED was pulsed for 4 milliseconds. We used the MSP430 to “walk the pulse” along the odd and even fields until the LED appeared in the camera image, thus determining when the shutter was open.

After determining when the shutter was open, we constructed a laser pulsing and hardware interlock safety circuit to safely pulse the laser. The circuit consists of an LM1881 Video Sync Separator, a 74123 retriggerable monostable multivibrator, a 7408 AND gate, and a Texas Instruments MSP430 microcontroller. The hardware interlock circuitry assures that the laser is pulsed for the appropriate duration. We made calculations using the American National Standard for Safe Use of Lasers to determine the maximum pulsed power a continuous wave laser can have for a given pulse

duration and still be eye safe at the laser exit. We based these calculations on the wavelength of the laser, the laser geometry, the pulse duration, and the pulse repetition frequency.

We showed that we can use polarization successfully to improve the signal-to-clutter ratio in a structured lighting system for use in outdoor applications. For indoor applications, considering the surfaces analyzed, the technique seems to have minimal effect. This is due to the large amount of depolarization that takes place in the laser light after reflection, and to the background light remaining unpolarized after reflection. Although the improvement in the signal-to-noise ratio may not be sufficient, the combination of polarization with spectral estimation and pulse-width modulated laser synchronization is sufficient. The implementation of such a system is also cost effective. To create a system that can perform in real time on a moving platform, one of two solutions is possible. The first solution is to register the images from three separate synchronized cameras. The second option is to employ Canon's 3CCD color camera technology. This technology uses a prism beam splitter to divide the primary optical path into three separate paths with a filter in each path that selects the desired spectral component. We would use our own filters.

Laser pulsing also provided the added benefit of reducing power consumption and size, which are paramount for mobile platforms where space and power are both issues.

# Biophysics of Biological Weapon Warhead Defeat with a Kinetic Interceptor

*67030*

*L. E. Larsen, S. A. MacNab, W. D. Reinhart, R. P. Toth, T. J. Vogler, A. Allen, D. D. Burchard, T. F. Thornhill III*

## Abstract

Development of an effective missile defense is a high priority national security objective. In addition, there is increasing concern about the spread of chemical and biological agents. If a missile carries a biological warfare (BW) agent, intercepting it may still result in a dangerous scattering of the infectious agent on military and civilian personnel. Thus, the intercepting vehicle must also kill the BW agent. Most mechanisms (radiation, etc.) are incapable of doing so in the brief interval before the agent is scattered. To accomplish the goal of killing before scatter, we propose to explore the biophysics of cell membrane disruption of BW agents by shock waves from a high-speed projectile.

High-fidelity bacterial simulants of wet plague (Gram-negative), germinated anthrax (Gram-positive), and sporulated anthrax will be loaded in a specially designed containment fixture and shocked by a projectile. The simulants will then be extracted, and the survival of the bacteria vs. impact velocity will be determined from the growth of colony forming units. Pressure and temperature will be derived from known equations of state and the projectile velocity. We expect that different classes of bacteria will differ in their resistance to shock loading. The spore form should be most resistant, then Gram-positive vegetative forms, then Gram-negative vegetative forms. There is some evidence, though, that shock loading may cause spores to clump, making them less infectious.

A systematic study of agent survival vs. pressure for kPa, MPa, and GPa pressures will be the first of its kind and will be directly applicable to the design of intercept vehicles and simulations of intercept events. These results may also be important in the destruction of agent stockpiles. The technology from this project will give Sandia a unique capability for the study of chemical and biological agents.

## Accomplishments

We studied high-pressure shock loading as a means to kill bioagents. We designed and fabricated capsules for shock loading the microbial samples. We provided control and experimental capsules for handling and transport effects. These capsules contained the samples. Secondary and tertiary containment were provided in case of leakage. We monitored leakage in the experimental capsules with a dye. We used equation of state models to calculate the initial and first reverberation (all others were attenuated).

We investigated a form of *Yersinia pestis*, which is responsible for plague, and *Bacillus cereus*, a close relative of *Bacillus anthracis*. Bacterial samples in capsules were shock loaded with a gas gun to pressures from 1.1 to 3.3 GPa. Following shock loading, we recovered the samples and assessed bacterial survival via serial dilution and growth of colony forming units. We found both microbes to have a threshold pressure above which a very high percentage of cells were killed due to rupture of their cell walls. Surprisingly, the threshold pressure was somewhat higher for *Y. pestis* than for *B. cereus*, despite the more robust cell wall of the latter. These results represent the first systematic and well controlled study of the susceptibility of bacteria to shock loading, and should provide valuable data for shock defeat of bioagents.

## Refereed

T.J. Vogler, T.F. Thornhill, W.D. Reinhart, L.C. Chhabildas, A. Allen, D.D. Burchard, S. MacNab, R.P. Toth, and L.E. Larsen, “Shock Kill of Biological Agents,” presented and in *Proceedings of the Missile and Warhead Conference*, Monterey, CA, August 2005, CD-ROM.

# Weaponization of Thermobaric Explosives

*67032*

*A. M. Renlund, R. J. Pahl, M. J. Kaneshige, M. R. Baer, M. A. Cooper*

## Abstract

There is a growing need in the United States to develop energetic materials (EMs) that, when integrated into munitions, offer advantages of enhanced lethality against a variety of targets and increase the firepower of expensive precision delivery systems. Lighter and/or less bulky munitions significantly impact the logistics burden on military actions. However, increased energy density of the EM described either in energy per volume or energy per mass is only one consideration. The additional requirement is that the energy be released in an appropriate time domain to allow optimized coupling to the target.

For enhanced lethal effects, the energy must be released either in the detonation reaction zone or early enough in the expansion so that it couples to impulse loading or sustained high temperatures. In thermobaric explosives (TBXs), the later energy release from metal combustion is postulated to play a dominant role in enhancing lethality against soft targets like buildings, materials, and personnel in tunnels. This project will focus on specific issues related to paste materials (consisting of flake aluminum, the solid explosive RDX, the liquid monopropellant isopropyl nitrate, and a gelling agent) in warhead-like configurations. We will perform simulations and experiments to assess material survivability in severe impact conditions. We will examine the material dispersal of paste explosives to measure the distance fuel is carried from the charge center, and compare this performance with solid aluminized explosives. We will examine the coupling of early explosive performance to warhead case rupture for optimal dispersal and TBX lethality. A key technical element is to evaluate the effects of material casing on dispersal and, ultimately, on the coupling of lethal energy to a target.

## Accomplishments

We conducted open-field tests of up to 20 kg on the TBX to monitor material dispersal, separation of dispersed phase materials, formation of large scale jets and turbulent structures, formation of secondary shocks, and the deceleration of gas expansion products. We also conducted comparison tests on Composition C-4 at similar charge masses. C-4 is a conventional high explosive that, unlike TBX, contains no aluminum. We incorporated performance and scaling behavior into an initial model to simulate detonics and dispersal. We participated in a series of multinational tests on TBX materials, including spherical charges designed by Sandia. These open-field tests included both plastic and metal-cased explosives and different charge masses.

To enhance mixing, we tested plastic-cased TBX charges of up to 3 kg in a suitable steel vessel that provided uncluttered internal geometry and shock focusing. We tested both TBX and C-4 for comparison, using a suite of diagnostics that included chamber pressure, strain, and temperature. We measured a significant temperature rise for the TBX material.

We tested a series of TBX formulations, varying the explosive powder, aluminum particle size, and the addition of oxidizer inside the 1-kg-limit chamber. Diagnostics included high speed imaging, both free-field and reflected pressure gauges, impulse gauges, and multichannel pyrometry. We conducted tests in both air and nitrogen atmospheres. Post-test analysis of the aluminum particulate showed that it was fully oxidized and no nitrides were observed in the reactions in a nitrogen atmosphere. We believe this indicates that the aluminum reacts within the detonation products, and that secondary combustion with the air is due to other fuel species from the detonation.

We designed and conducted an initial series of tests on steel-confined charges. Preliminary model calculations show that thicker, heavy cases enhance mixing by constraining and folding by multiple shocks.

## Other Communications

M.A. Cooper, M.J. Kaneshige, A.M. Renlund, and R.J. Pahl, “Thermobaric Experiments Toward a Physical Understanding of Enhanced Blast Explosive Performance,” presented and in *Proceedings of JANNAF 40th CS, 28th APS, 22nd PSHS and 4th MSS Joint Meeting*, Charleston, SC, June 2005, CD-ROM.

M.R. Baer, R.G. Schmitt, E.S. Hertel, and P.E. DesJardin, “Modeling Enhanced Blast Explosives Using a

Multiphase Mixture Approach," presented and in *Proceedings of JANNAF 40th CS, 28th APS, 22nd PSHS and 4th MSS Joint Meeting*, Charleston, SC, June 2005, CD-ROM.

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# Development of an Enterprise-Scale Agent-Based Autonomic Logistics Simulation Model

*67033*

*D. A. Schoenwald, W. E. Hart, L. P. Swiler, B. Chenoweth Creel, B. M. Thompson, J. Watson*

## Abstract

This project focused on the development of a general purpose enterprise-scale logistics support system modeling capability. The model will incorporate various optimization strategies that will assist users in meeting performance metrics, managing risk, and minimizing costs. Since the "best" logistics support system will change over time, both the model and the optimization capabilities must be capable of addressing the time dependent nature of the problem. One of the biggest factors driving the need for logistics system models is the large scale nature of many military and commercial systems and the associated costs. These systems typically contain thousands of parts from hundreds of different suppliers, all with different manufacturing schedules.

Multiplying the number of parts per system by the number of systems that may be deployed over the production cycle, one can see that potentially millions of parts may need to be tracked worldwide. Therefore, this model will be capable of handling such a large volume of parts and systems and updating their status regularly. This model will not only be able to track such data, but it will be able to analyze the data and quickly deliver reliable information to support the design and operation of the logistics system to deal with unexpected events (war, labor disputes, natural disasters, etc.). This implies the ability to do fast and dependable optimization of the model for a variety of design questions and "what if" scenarios. Finally, the model will be accurate in its portrayal of the status and location of all systems and resources. It will also be accurate in its prediction of resource usage and equipment performance metrics.

## Accomplishments

During FY 2005, the project team was successful in several key tasks for fielding a realistic weapons system enterprise prototype. These tasks primarily focused on building a general agent-based weapons logistics model that is not tied to any particular application but is capable of simulating a range of applications with only input file modifications. First, we finished the basic architecture design for the agent-based model. This architecture leverages the work (including parallelization and user interfaces) of the NABLE (NISAC Agent-Based Laboratory for Economics) team. Second, we coded the software in C++ to run a small scale test problem. Third, we designed a small scale test problem consisting of a basic military aircraft supply and repair chain. Fourth, we completed much of the parallelization effort with only the load balancing remaining. Finally, we finished initial work on the optimization strategy. Here the two biggest subtasks were to determine several cost criteria and a general strategy to pursue. The cost criteria will include a combination of inventory and transportation costs along with maintenance costs for a certain level of weapons system availability. The general strategy will be to use an analytic model as an approximation of the full agent-based model. We will use a mixed integer linear programming approach on the analytic model for the global optimization.

## Refereed

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D.A. Schoenwald, D.C. Barton, M.A. Ehlen, "An Agent-Based Simulation Laboratory for Economics and Infrastructure Interdependency," presented and in *Proceedings of 2004 American Control Conference*, Boston, MA, July 2004, CD-ROM.

# Mobile Node Authentication in a Wireless Ad Hoc Environment

*67034*

*J. T. Michalski, M. D. Torgerson, B. P. Van Leeuwen, A. J. Lanzone*

## Abstract

In hostile ad hoc wireless communication environments, such as battlefield networks, end-node authentication is critical. In a wired infrastructure, this authentication service is typically facilitated by a centrally located "authentication certificate generator" such as a certificate authority (CA) server. This centralized approach is ill-suited to meet the needs of mobile ad hoc networks, such as those required by military systems, because of the unpredictable connectivity and dynamic routing. There is a need for a secure and robust approach to mobile node authentication.

Current mechanisms either assign a preshared key (shared by all participating parties) or require that each node retain a collection of individual keys that are used to communicate with other individual nodes. Both of these approaches have scalability issues and allow a single compromised node to jeopardize the entire mobile node community.

In this project, we propose replacing the centralized CA with a distributed CA whose responsibilities are shared between a set of select network nodes. To that end, we developed a protocol that relies on threshold cryptography to perform the fundamental CA duties in a distributed fashion. The protocol is meticulously defined and is implemented in a series of detailed models. Using these models, we created mobile wireless scenarios on a communication simulator to test the protocol in an operational environment and to gather statistics on its scalability and performance.

## Accomplishments

The team developed some associated wireless attributes that provide an efficient and streamlined approach in supporting the authentication process. Some of the attributes we developed follow:

Group-cast certificate request: The group-cast method of information request is more scalable and efficient than broadcast forms of the same request. A broadcast request requires each node to receive the broadcast process and forward the request, even when the receiving node does not have the capability of providing part of the answer. With the group-cast method, only nodes that are associated with the "group" address are asked to process and potentially forward the request.

Selectable outreach: How far the initial request is propagated is selectable, as described in the following modes:

(1) Default Mode: In this mode, the initial reach of the request can be limited to a user-defined broadcast domain. This allows for a very rapid response to an initial request.

(2) Sparse Certificate Authority Scenario Mode: In this mode, the reach capability can be extended to encompass multiple broadcast domains over a widely dispersed geographical area.

(3) Redundant and Stale Request Detection: The design of the protocol provides a means for a receiving node to determine whether the incoming certificate request is an original or redundant request; all redundant requests are discarded. The protocol also provides a means for mobile nodes forwarding requests to "time-out" a request after a preselected amount of time.

# Novel Processing, Affordable Motion Compensation, and Mode Multiplexing for Miniaturized Synthetic Aperture Radar

*67035*

*G. R. Sloan, A. D. Sweet, T. J. Kim, A. W. Doerry, M. W. Holzrichter, D. F. Dubbert, D. C. Sprauer, P. G. Ortiz*

## Abstract

Sandia recently demonstrated a revolutionary synthetic aperture radar (SAR) that achieves a 5x size/weight/cost reduction with no degradation in image quality or resolution. The miniSAR development is in response to the critical need for miniaturized, affordable SAR for both tactical unmanned aerial vehicle based reconnaissance and all-weather global positioning system denied precision weapon guidance. This project helped facilitate the success of phase one of miniSAR by developing a revolutionary software architecture (radar control, motion measurement, and image formation) that successfully exploits the new miniSAR hardware.

The proposed research and development for this project follows:

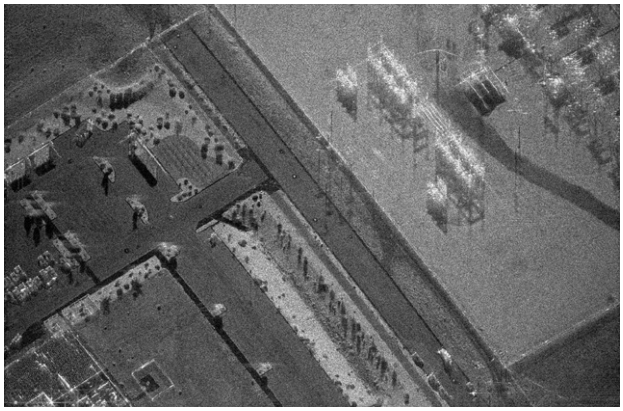
(1) develop and implement a revolutionary, highly modular, easily modified software architecture for miniSAR;

(2) study and develop optimal miniSAR image formation algorithms and approaches (for both processor and gate array based implementations);

(3) develop key sub-blocks of field programmable gate array (FPGA) based image formation hardware; and

(4) investigate the viability of using a smaller, lower-cost IMU (inertial measurement unit) by enhancing Sandia's autofocus algorithm and by employing Sandia-developed IMU rotation concepts.

At the conclusion of FY 2005, tasks one and two are largely complete, and we have made significant progress on the other tasks. The latest tests of the in-development FPGA-based image engine indicate that it will provide a 60x improvement in processing speed over the general purpose processor currently used in miniSAR. (This, in turn, facilitates new radar modes and capabilities that are important to national security applications.) The effort to adapt lower grade IMUs to fine resolution SAR will directly lead to significant miniaturization and cost reduction.



*Real time SAR image (power substation) made by miniSAR radar.*

## Accomplishments

- Software Architecture:

We completed a novel modular radar software architecture and integrated it into miniSAR. Some of the innovative features include the following: (1) the software achieves new levels of independence from the radar hardware, easing the migration to future designs/enhancements; (2) the unprecedented level of modularity will make it much easier to add new radar modes in the future; and (3) the communication between the operator interface and embedded routines achieves unprecedented flexibility and robustness. In addition, we developed a novel polar format image formation algorithm that provides improved efficiency and speed. This algorithm replaces the previous chirp-Z transform with a more computationally efficient post-azimuth-FFT (fast Fourier transformation) interpolation. We added an initial version of this algorithm as an option to the miniSAR image formation software; it was successfully verified in flight testing. When fully optimized, it may provide up to a 50 percent reduction in processing time. We filed an invention disclosure for this concept. After weeks of flight tests and hundreds of images, we determined that the overall software architecture is exceeding expectations.

- FPGA-based Image Engine:

The FPGA-based image engine effort includes the implementation of both a polar format algorithm and an autofocus algorithm. The image formation and autofocus blocks are each composed of several sub-blocks that we are developing and testing independently. Recent benchmarks indicate that the image engine will be 60x faster than the general purpose processor currently used in miniSAR. The following image formation tasks were completed in FY 2005:

(1) We completed the design and modeling of the novel FPGA-compatible image formation algorithm.

(2) We completed the quantization analysis for each subcircuit block (for both the image former and autofocus algorithms).

(3) We completed the VHDL (virtual hardware description language) mapping of the image formation algorithm.

(4) We created a Simulink/Sysgen-based model of the entire autofocus design. We successfully used simulations to focus actual radar images.

(5) We successfully validated about 75 percent of the sub-blocks in FPGA evaluation boards.

- Motion Measurement:

We further evaluated the enhanced autofocus algorithm developed in FY 2004. This algorithm is one method we are pursuing to facilitate the use of lower quality IMUs (smaller, cheaper). Outside of this project, this algorithm was successfully used to process ultrahigh resolution images from a new Sandia radar. Additionally, we are currently working with other Sandia projects to evaluate the beneficial effect of periodic rotation (carouselling) on lower grade IMUs (specifically, the Honeywell MEMS-based HG1930). Initial results look promising.

## Other Communications

G.D. Martin, A.W. Doerry, and M.W. Holzrichter, “A Novel Polar Format Algorithm for SAR Images Utilizing Post Azimuth Transform Interpolation,” Sandia Report, SAND2005-5510, Albuquerque, NM, 2005.

R. Hurley, “Taking Reconnaissance to Another Level,” presented at the IEEE MTT-S International Microwave Symposium, See-Through Radar Workshop, Long Beach, CA, June 2005.

G. Sloan, “MiniSAR—Development Status and Description,” presented at the Department of Energy PDP TIE Conference, Albuquerque, NM, April 2005.

# Coilgun Technology Demonstration Testbed

*67036*

*R. J. Kaye, G. A. Mann*

## Abstract

Induction coilguns, which use electromagnetic force without a sliding electrical contact instead of chemical propellant, are capable of launching projectiles of interest to national security. Technical issues that remain include (1) lifetime of coils that make up the gun barrel, (2) integrity of the coil's mechanical structure, (3) demonstration of repetitively fired coils with thermal management, and (4) robustness of fire/control systems. The objective of this project was to design a coilgun coil capable of repetitive firing, and then test that design at loads consistent with a Department of Defense (DoD) application for lifetime data. This work is in collaboration with the National High Magnetic Field Laboratory (NHMFL) at Florida State University and with Los Alamos National Laboratory.

The key accomplishment in FY 2004 was the development of a 120 mm bore, nested-helix coil design with active heat transfer mechanisms for repetitive operation in a coilgun at the mechanical, electrical, and thermal stresses necessary for application in the Army's Future Combat System. Key accomplishments of FY 2005 include the establishment of a construction process based on independent wire-in-conduit helical winding layers to reduce cost and open fabrication options, the development of a process for weld-joining of conductors of multiple independent winding layers, construction of wire-in-conduit windings applicable for thermal testing, and continuous testing of a wire-in-conduit winding to demonstrate the heat transfer capability of the concept.

## Accomplishments

In FY 2005, we focused on the detailed design and fabrication of prototype coils for testing:

(1) The fabrication process for a multilayer coil is now more feasible and cost effective as a result of changing from a single wire-in-conduit for multiple layers to joined independent layers. This change will facilitate the acquisition of conduit and permit use of preformed Zylon® poly-p-phenylenebenzobisoxazole (PBO) reinforcing shells to simplify assembly and reduce cost. The first experience winding a multiple layer coil from a single long conduit demonstrated the need to split the conduit for coolant access between layers, defeating the benefit of a continuous conduit. We modified tooling and fabrication techniques to accommodate this change.

(2) We established a tungsten inert gas (TIG) wire welding method for joining wire between winding layers during coil assembly. Welded wire samples show very good quality joints.

(3) We established concepts for the joining of conduit, but did not complete fabrication and testing. This remains the greatest issue in completion of a multilayer coil structure.

(4) We completed 10 ft long electrically insulated wire-in-conduit assemblies and demonstrated the ability to control wire position in the conduit while winding into a coil.

(5) With test windings, we demonstrated the ability to control conduit geometry tolerance to permit joining of multiple, independent layers in a coil.

(6) We did not complete fully reinforced prototype coils, but we prepared a wire-in-conduit winding of the first coil layer for test and evaluation of heat transfer properties. Heat transfer is the most important property of the design that had not been independently tested because it requires completed, wound conduit structures wherein the as-built geometry governs performance.

(7) We constructed a coil testbed to evaluate the heat transfer coefficient of the wire-in-conduit winding by continuously energizing the winding to achieve the highest conductor temperature possible within the boiling limit of the coolant. The thermally insulated coil was instrumented for pressure, temperature, flow, current, and voltage measurements. We observed that the heat transfer of the design appears to meet or exceed the expectations calculated during the design. We must add additional temperature probes to obtain local heat transfer coefficients along the length of the winding and to precisely compare to thermal models.

(8) We pursued the characterization of new composite poly(pyridobisimidazole) (PIPD) fibers.

(9) We demonstrated the performance of the key components of the repetitive coil design: sufficient heat transfer from wire to coolant, mechanical strength of

the PBO fiber composite, and mechanical/electrical strength of the Kapton/Zylon insulation. Integration of compact coolant feeds and assembly of multiple layers with composite reinforcement shells are the remaining issues to be addressed prior to repetitive demonstration of the integrity of a complete coil.

# Enhanced Perception for Remote 3-D Mapping of Unknown Indoor and Outdoor Environments

*67037*

*C. L. Lewis, G. A. Laguna, C. Q. Little, F. Rothganger*

## Abstract

A robotic vehicle's ability to simultaneously localize and accurately map its surroundings is crucial to its autonomy and to an enabling technology for future minimally manned systems applications. In addition to being critical for the robot's autonomy, localization and mapping provide essential feedback to the operator for area intelligence, surveillance, reconnaissance, and remote crisis response. Robotic three-dimensional (3-D) mapping technology provides enhanced situational awareness by permitting a virtual "fly through" of denied environments. Simultaneous localization and mapping (SLAM) has seen an explosion of interest throughout the robotics community. Most implementations use probabilistic state estimation techniques to combine measurements from multiple intrinsic and extrinsic sources. Laser range finders and video cameras are common extrinsic sensors.

The processing load of matching data from nodding laser scanners or video cameras to extrinsically estimate motion is prohibitive. Therefore, a few easily identified features are tracked. For instance, because David Lowe's scale invariant feature tracking (SIFT) algorithm identifies landmarks in video images that are easy to track, a number of implementations rely on it. Other algorithms pick out vertical and horizontal edges from laser scanner data to track. With either of these methods, the resulting maps miss obstacles because they do not contain either visual or special features that stand out. In our work, we use both range and video data to select both high contrast and shapely features. Furthermore, the entire colorized range image is processed to form the navigational map. The contrasting and shapely landmarks are used only to derive the extrinsic motion measurement needed for localization. We will develop maximum likelihood models that represent the robot's perception of its environment. The resulting map is a terrain model overlaid with objects perceived by the robot. Dynamic objects will be detected and tracked, but not added to the map.

## Accomplishments

To enable 3-D scanning, we developed a nodding device. Along with the scanner, we mounted a video camera on the nodding device. We developed calibration algorithms to create 3-D colorized range images. We defined a feature descriptor that locates shapely features with high contrast. We will track these features using SLAM, keeping only the localization data. To generate maps, we are developing algorithms to decimate the SLAM registered-colorized range images and to generate 3-D maps on rugged terrain.

By teaming with LDRD Project 79831, "Design Tools for Complex Dynamic Security Systems," we were able to add a nodding device to an AutoNav platform (HAGAR) and update its software to be compatible with the SWARM code. HAGAR now runs the Linux operating system, communicates with the SWARM base station, has an auxiliary differential global positioning system (DGPS), and can create maps. We can now use HAGAR as part of an adaptive security system demonstration where the nodding laser scanner and video system first generate a map of the facility. HAGAR will patrol the area and be available to intercept intrusions. Teaming helps disseminate our mapping capabilities and also ensures we remain grounded in practical applications.

In a parallel effort, we investigated the iterative closest point algorithm for its ability to perform localization in maps. We developed a method for discerning the sensitivity of a bulk scan match. Sensitivity measures are critical for characterizing the quality of a derived extrinsic motion measurement. For example, consider matching two laser scans of a flat plane. The transform that yields the best match between the scans determines six extrinsic location measurements. The quality of the match is independent of three of the derived variables and thus provides no information with respect to them.

## Refereed

C. Little and R. Peters, “Simulated Mobile Self-Location Using 3D Range Sensing and an A-Priori Map,” presented and in *Proceedings of the 2005 IEEE International Conference on Robotics and Automation*, Barcelona, Spain, April 2005, pp. 1471–1476.

# A Modular Micromagnetic Accelerometer in Support of the mTalon Vision

*67038*

*R. A. Kellogg, J. K. Brotz, R. S. Goeke, J. A. Romero, B. Jokiel Jr., T. Lemp*

## Abstract

Advanced seismic sensors can provide monitoring capabilities for defense applications, including underground facilities detection and battlefield situational awareness. The sensors can also provide an imaging capability of the earth's interior for identifying geologic structures containing energy reserves. New seismic sensors are needed, however, because existing devices are expensive, large, fragile, or lack the sensitivity in the frequency bands of interest.

This project focuses on the development of small, low power, rugged, and inexpensive seismic sensors using Sandia's microelectromechanical system (MEMS) technologies, including the bulk micromachining of Si and ferromagnetic materials integration. As such, we examine the fabrication, processing, and assembly methods for a modular micromagnetic accelerometer based on giant magnetoresistive (GMR) magnetometer technology. The realization of the micromagnetic accelerometer depends on the integration of an inertial sense mass element and permanent magnet with a magnetic based, high-resolution position detector. The inertial sense mass includes a micromachined Si spring/mass system that has low damping and resistance to off-axis motion. The position sensor includes a GMR sensor array designed for detecting less than 1 picometer shifts in magnetic field induced by the motion of the sense mass and magnet. To attain acceptable low noise performance of the device, the sense mass, magnet, and GMR detector components must operate in a near vacuum sustained by the packaging. Many engineering challenges and development issues were associated with the design, fabrication, and packaging of the micromagnetic accelerometer.

We successfully produced the Si sense mass and spring elements on the wafer scale using deep reactive ion etching. However, the insertion of magnet material on the bulk scale was problematic. We eventually accomplished magnet integration using time consuming hand installation methods. After much effort in developing a die alignment and low temperature solder bonding method, we only assembled one GMR sensor die and Si shuttle. We did not realize successful packaging and testing before the project's conclusion. Additional process development is needed before the micromagnetic accelerometer may be successfully realized.

## Accomplishments

The FY 2005 goal for the micromagnetic accelerometer project was to realize a tested prototype. Toward this end, we focused on materials integration, assembly, electronics, and test system development. We previously fabricated the accelerometer's Si sense mass (350 micrometers thick) with 100 micrometer-wide slots to accept magnet material for position sensing with GMR sensors. We made attempts to integrate bonded NdFeB magnet material using high-force pressing and vibratory agitation, but they proved unsuccessful due to voiding and Si wafer breakage. As an alternative, we developed hand calendaring methods using methanol solvent for incorporating epoxy and magnet powder into the slots. For detecting sense-mass position, GMR sensor die must be accurately aligned to within 1 micrometer in three dimensions and bonded to the Si spring-mass die. We tested low temperature die-bonding methods to establish assembly parameters and evaluate bonding strengths. We joined the Si shuttle and GMR sensor die using a 450 angstrom thick chromium adhesion film, followed by a 2000 angstrom thick gold film for bonding to a tin-indium eutectic solder thin film multilayer.

To provide a vacuum environment and facilitate low noise accelerometer operation, we tested low temperature packaging methods using low outgassing epoxies. We developed support electronics to measure the output signal from the GMR sensors. We designed and fabricated printed circuit boards to accommodate the accelerometer mounting with circuitry for electrical testing. We developed a multiplexed test system to analyze an array of sensor and magnet dimension combinations. Additionally, a chip-scale lock-in amplifier was adapted to permit low level accelerometer signal detection with 100 dB of noise rejection. To facilitate accelerometer testing, we designed and fabricated a magnetically shielded enclosure and rigid fixture. We also assembled a micro-g test bed, including an electrodynamic shaker and amplifier on a granite slab and iso-

lation table, to provide a low vibrational, acoustic, electrical, and magnetic noise environment. Process development issues confounded the successful final assembly and packaging of the micromagnetic accelerometer. Although we did not test a prototype accelerometer, we learned much along the way about Si and magnetic materials integration as well as low temperature, high-precision die alignment and bonding, and low noise electronics.

# MICROFUZE Integration

*67039*

*A. W. Roesler, M. T. Dugger, D. J. Frew, B. M. Melof, T. E. Buchheit, R. McEntire, A. S. Tappan, S. M. Wooden, E. J. Welle*

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# Analysis of Technology Impacts on Operations in Complex Environments

*67041*

*M. J. McDonald, F. J. Oppel III, E. P. Parker, S. Tucker, B. P. Van Leeuwen, K. J. Page*

## Abstract

The main product of this project is a unique capability to assess technology impacts in urban environments. While additional work is needed, technology developers as well as military and security technology decision makers are already finding the tools developed in this project to be contributing to their success.

When we initiated this project, 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. This project made significant and credible progress in addressing those issues.

A key aspect of this effort is the actual performance of technology impact analyses addressing realistic scenarios to identify needs and to drive new model development. As a result, this project developed new technologies of integration and identified several emerging Sandia codes for inclusion. We also began to extend and integrate 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. We are using existing urban and Department of Energy (DOE) facility terrain models for analyses.

## Accomplishments

We completed all planned tasks, met all planned milestones, and are ahead of schedule on the remaining tasks and milestones.

We made significant progress on several technical and organizational fronts. The project team transitioned all its models to a completely rewritten release of the Umbra modeling framework and a new system of systems representation. We also completely re-factored our sensor models to better address complex geometries, developed new models to address people and combat, and developed new communications algorithms to address command and control information architectures. In addition, we built a variety of high resolution terrains to support experimentation.

Work in understanding user needs in security allowed us to help define and then to leverage results from two significant complex systems analysis efforts. The first systems analysis contributed to the success of the Virtual Perimeter System Grand Challenge LDRD project. The second led to a DOE funded effort in generative modeling for security needs assessments. These efforts place a high emphasis on scenario development that we leveraged to allow more emphasis on modeling and technologies of integration. In addition, technologies developed in this project are being used for intelligent systems and robotics, and other applications such as development of advanced weapon trainers, analysis of border security operations, simulation of nonproliferation technology test sites, and the analysis of material transport.

A highlight of the work this year was the completion of a second “summer study” that accomplished an unprecedented level of model integration and addressed issues that have great significance to both emerging nuclear weapon security concepts and new military battle environments. This study environment combined several models including two distinct sensor network models; a command and control network model; individual combatant models; fixed wing, rotary wing, and hovering unmanned air vehicle models; and precision missiles, weapons, barriers, and background traffic models. The command and control as well as the combatant models are driven with a new human behavior engine.

## Other Communications

M.J. McDonald, “Umbra’s System Representation,” Sandia Report SAND2005-3819, Albuquerque, NM, 2005.

# CBW Cloud Knockdown and Neutralization

*67042*

*R. G. Betty, B. L. Levin, D. A. Lucero, J. E. Brockmann, M. D. Tucker*

## Abstract

One technique for the dispersal of CBW (chemical and biological warfare) agents is to create a cloud of active material that would drift to affect intended target areas such as military bases, battlefields, or urban areas. The goals of this project were to determine feasibility and investigate methods of aerosolized CBW cloud knockdown and neutralization, particularly in support of outdoor, fixed-site force protection.

By successful combination of Sandia expertise in aerosol science and DF-200 CBW agent decontamination technology, we demonstrated proof of concept of charged decontaminant sprays for neutralization and mitigation of aerosolized CBW agent simulants. Of significance is the potential application of this fundamental technology to numerous applications, including mitigation and neutralization of releases arising during chemical demilitarization operations, protection of chemical industry facilities, public venues such as subways, and fixed-site protection.

## Accomplishments

We performed spray chamber tests using appropriate chemical and biological agent simulants necessary for model development, validation, and calibration. We commenced aerosol chamber testing. Preliminary chamber testing indicated knockdown and neutralization of chemical simulant agent concentrations ranging from 2 to 4 orders of magnitude. We collected results following use of standard DF-200 deployed from electrostatic spray nozzles (ESS Maxcharge™) in the charged state. Initial results demonstrated the potential efficiency of charged mitigation sprays in neutralizing aerosolized agents. Aerosol chamber testing continued throughout the fiscal year.

Using threat agent scenarios, we performed Gaussian modeling to predict agent plume exposure concentrations at 2, 5, and 10 km downwind of an agent release. We then used peak plume exposure concentrations as target simulant concentrations during aerosol test chamber testing.

We demonstrated rapid and effective knockdown and neutralization of chemical and biological simulants. We released an aerosolized cloud of a G-agent simulant (diphenyl chlorophosphate) at a concentration of 3.2 g/m$^3$ in an 8' x 8' x 8' test chamber. We mixed the cloud using a series of fans in the test chamber for a period of 50 minutes. After that time, we deployed a spray of DF-200 for one minute through a series of nine ESSs located in an array at the top of the test chamber (the air pressure supplied to the nozzles was approximately 80 psig). The total volume of DF-200 spray deployed in the chamber was 2 L, and the concentration of DF-200 in the chamber was approximately 138 g/m$^3$, making the challenge ratio (decon:simulant) approximately 40:1. We collected the G-agent simulant by aerosol sampling. We determined the simulant concentration in the chamber by gas chromatography immediately after the end of the spray period and again at 15 and 30 minutes after the end of the spray period. The results demonstrated nearly 4 LOG knockdown and neutralization of the simulant immediately after we stopped the spray. This decrease in agent simulant concentration is in excess of the 1.5–2 orders of magnitude needed to bring potential chemical agent exposures below the $LCt_{50}$. As a comparison, starting with nearly 2 orders of magnitude greater concentration, data indicate approximately 4 orders of knockdown and neutralization of the G-agent surrogate accomplished using 90% less formulation.

*Video of aerosol test chamber experimental process.*

Using a similar test matrix, we also demonstrated knockdown and neutralization of *Bacillus globigii (atrophaeus)*, a surrogate for anthrax. We determined initial chamber aerosol samples to contain 5.1 LOG

CFU/L of *B. atrophaeus*. *B. atrophaeus* was not detected immediately or after 15 minutes following a 1 minute charged DF-200 spray, indicating a knockdown and neutralization of greater than 5 orders of magnitude. A decrease in 4 orders of magnitude is needed to bring biological agent peak exposure concentration below the $ICt_{50}$. Additional testing using a charged DF-200 spray density of 92 $g/m^3$ demonstrated a 5 LOG knockdown and neutralization of the anthrax simulant; a 46 $g/m^3$ charged DF-200 spray density demonstrated 4 LOG immediate knockdown and neutralization. We also directed experimental efforts at knockdown and neutralization of various other agent threats and assessed variables to the spray system such as ESS nozzle charge, spray droplet size, decontamination formula, and spray duration.

We performed assessments of various CBW cloud knockdown and decontamination deployment concepts for force protection at a stand-alone facility such as an airbase. Knockdown approaches we considered include ground based knockdown systems, fan assisted systems, and aircraft knockdown. We developed optimum deployment concepts for subway protection and chemical demilitarization based on the feasibility and efficiency of achieving cloud knockdown and decontamination.

# Adaptive Force Feedback Surface Control

*67044*

*D. K. Kholwadwala, J. W. Wheeler, G. L. Benavides, B. Hassan, P. C. Galambos, D. L. Keese, B. R. Rohrer*

## Abstract

We are developing a novel massively parallel haptic device to sense and control surfaces. These devices contain independent fluid-filled bladders that act as both sensor and actuator. An integrated microelectromechanical systems (MEMS) pressure sensor and MEMS valve determine the pressure applied to a given bladder and respond by regulating the fluid flow to the bladders. When placed in a large array, these devices are capable of creating and controlling arbitrary surface shapes.

Two major accomplishments are the development of a prototype for concept validation and the refinement of MEMS component designs. Using commercially available parts, we developed a prototype for testing that was controlled by a personal computer (PC) using Matlab. We improved MEMS pressure transducer and valve designs to provide more functional and reliable components. We also developed improved microsystems packaging techniques.

## Accomplishments

We met two major project goals this year.

First, we created a prototype adaptive surface using commercially available components. The purpose of the prototype was to validate the concept and test control systems. We designed and fabricated custom silicon-rubber balloons that deflect primarily along their major axis. We also fabricated custom mounting components for 40 balloon units. We purchased fluidic components (e.g., valves and pressure transducers). We connected each balloon unit to a pressurized air source through an inlet valve and to the atmosphere with an outlet valve. The pressure transducers monitored the pressure in each balloon. We assembled the system and connected it to a PC for real time control. We wrote Matlab scripts to regulate the pressure in each balloon in a closed-loop fashion. We used the prototype to validate the adaptive surface concept by moving a soccer ball across the surface by selectively inflating and deflating balloons.

Second, we refined MEMS pressure transducer and valve designs to provide more functional and reliable components. Previous pressure transducers were functional but had a large voltage offset due to residual strain in the flexible membrane that limited the effective resolution of the devices. We added on-die trim resistors to balance this offset. These resistors can be selectively eliminated until the effective offset is near zero. Previous MEMS valve designs were nonfunctional due to sliding friction in the electromagnetic drives. Other designs could not be held closed against a pressure or were too leaky. The new valve designs use well characterized thermal actuators to move a slider valve. The new devices have lower leak rates and sufficient force capacity to overcome sliding friction.

We also developed improved packaging and microsystems integration processes during this project. We designed and built a new test setup for evaluating MEMS fluidic devices that is currently in use.

# High Capacity Earth Penetrator Instrumentation

*67046*

*B. J. Welch, R. R. Lockhart, M. Arviso, J. P. Ehasz*

## Abstract

In the past, our ability to collect critical engineering data during full scale earth penetrator tests did not meet the demands of the earth penetrator weapon systems. Specifically, sample rates were insufficient for the component level data needed, and the number of measurement signals supported was inadequate. To allow the development of these weapon systems to proceed with the necessary testing data, we developed a shock-hardened data recorder system with new approaches for measurement sampling and data storage capacity. Although with vastly greater capability than current designs, the instrumentation remained compact and extremely rugged.

Previous penetrator instrumentation designs recorded a maximum of 12 channels at 30,000 samples/second/channel with a 500 millisecond record time. Early robust nuclear earth penetrator tests indicated a need for more than 48 channels at rates of at least 100,000 samples/second. The high-capacity earth penetrator instrumentation meets these needs with a modularized system capable of recording 64 channels sampled at a rate of 150,000 samples/second. We tested the design in environments produced by the shock table, linear actuator, and gas gun.

## Accomplishments

This year we completed the flight form-factor board designs for the controller, acquisition, and capattery boards. We used the new board designs to build two "HiCapPen" units, a 19-channel recorder, and a 35-channel recorder.

The design underwent a flight qualification test series. This test series consisted of shock table testing, linear actuator testing, and a gas gun test. The tests stressed the units in environments ranging from 1000 to ~8000 Gs, with durations of 0.5 to 5 milliseconds. The test series concluded with a successful rocket sled test on the 10,000 foot rocket sled track. The sled was instrumented with both HiCapPen units (19- and 35-channels) to record measurements from over 20 sensors (there were measurement channels available for 48 sensors). This rocket sled test was the first instrumented test to record the entire sled ride from first motion to impact, roughly 10 seconds worth of data.

We also completed the automated tester (ATE) for the HiCapPen units. The ATE provides a mechanism to rapidly recertify units following test events and decreases the amount of time necessary for certifying new units.

# Systems Analysis of Networked Sensors

*67047*

*M. T. Oswald, P. E. Sholander, B. McDaniel, K. J. Smart, D. A. Wiegandt, D. Kilman, J. A. Rohwer, K. J. Mandke*

## Abstract

The goal of this project is to develop, analyze, and simulate robust, adaptable, and energy efficient wireless sensor network architectures. Our FY 2005 goals were to analyze and simulate adaptive, large scale, heterogeneous sensor networks. We previously defined the wireless sensor network architecture and simulation space. We completed significant work on analyzing and simulating wireless sensor networks for systems design.

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 subnetwork clusters to increase system throughput. The process steps included first order analysis, topology modeling, and network simulation of the distributed algorithms and communications.

The systems analysis process produced novel results. We completed an application specific analysis to define the problem within the network architecture and simulation space, and to provide baseline performance estimates. Next, we used topology analysis of clustering algorithms to narrow the design space. This analysis allowed us to rule out regions of the parameter space that will not produce working systems. Network simulations focused on the performance of distributed clustering algorithms over wireless communications links using models that included both bit error rate (BER) and fading effects.

Additional studies will support future project directions such as space based sensor networks. We obtained initial results on wireless sensor networks for space applications. We developed a communications model of rolling, divergent objects. The model outputs communications-link signal level traces as a function of time and statistical channel model parameters for inclusion in future analyses, models, and simulations. We also completed an overview of a cooperative communications techniques report in collaboration with the University of Texas. The concepts we explored will be considered for improving the performance of sensor networks for a space application.

## Accomplishments

Wireless sensor networks are routinely proposed as solutions for complex, distributed sensor applications. Current research addresses network designs based on standard architectures. The simulation spaces are well defined, and the results are impressive. The limitation of these well published algorithms is that current technology does not support efficient, low power hardware implementation of the signal processing algorithms and network protocols.

The goal of this project is to develop, analyze, and simulate robust, adaptable, and energy efficient wireless sensor network architectures. Our research is driven towards advanced sensor network architectures that are necessary for large scale, heterogeneous sensor networks. Improved systems analysis capability will support the future development of wireless sensor network for complex applications.

We considered wireless sensor network applications for analysis and simulation. We defined the concept of operation and design constraints for bomb damage assessment, space, and data exfiltration applications. We completed systems analysis of the data exfiltration and space wireless sensor networks.

The data exfiltration problem is to communicate data from 100 sensor nodes with delay and bandwidth constraints that preclude the use of a single satellite communications (SATCOM) relay. One solution is to use multiple ground based clusters with parallel SATCOM channels. In this manner, the network is broken into several smaller subnetworks that can exfiltrate all data within the time constraint. With this approach, the complexity is forced on assigning the clusters, cluster heads, and ground channels.

We performed a graph theoretic and topological analysis of the exfiltration application to refine baseline system performance analysis and eliminate design parameter combinations unlikely to provide a working system. The design parameters of interest were the clustering algorithm, node transmit power, and number of ground based communications channels.

Using network simulation results, we refined the graph theoretic and topological analysis. The network simulations show the performance of distributing the

clustering algorithm over sensor network nodes. The sensor network nodes make global cluster decisions based on local information. This analysis shows how the algorithm fails to converge when the unreliable nature of the wireless communications is included. We developed a method to improve the algorithm convergence and determined the design parameters likely to yield acceptable system performance.

The space sensor network application consists of communicating data from objects with time varying communications links. We developed a communications model for rolling, divergent, space objects. The model outputs communications link SNR (signal-to-noise ratio) traces as a function of time and statistical channel model parameters for inclusion in future analyses, models, and simulations. We compared the space application to the data exfiltration application. We also completed an investigation of cooperative communications techniques that will be considered for improving the performance of sensor networks for the space application.

## Refereed

B. McDaniel, M. Oswald, and P. Sholander, "Performance Analysis of Distributed Clustering Algorithms for Sensor Networks," to be published in *Proceedings of the 2005 Texas Wireless Symposium*.

M. Oswald, P. Sholander, B. McDaniel, J. Rohwer, and D. Kilman, "Data Exfiltration from Sensor Networks Subject to Delay, Connectivity and Frequency Re-Use Constraints," to be published in *Proceedings of the IEEE MILCOM Conference 2005*.

## Other Communications

K. Mandke and M. Oswald, "Overview of Cooperative Communications Techniques for Infrastructure and Ad Hoc Networks," Sandia Report SAND2005-5982, Albuquerque, NM, 2005.

# 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*

## Abstract

This project proposes to develop a system capable of completely characterizing the relative kinematic and dynamic properties of a deployed object from a remote platform with no contact or impact to any of the target objects. Specifically, the project proposes to advance three-dimensional (3-D) laser radar (ladar) to focus on applications related to National Missile Defense (NMD) concerns in space based deployments. The proposed system will have the ability to track multiple objects in the field of view. The technique we will use to acquire the data is scannerless range imaging (scannerless 3-D ladar) that uniquely provides high speed, high resolution 3-D imaging.

Sandia has significant experience and a leadership position with scannerless 3-D ladar. Sandia sensors are currently operating at the Laser Radar Range Facility of Eglin Air Force Base, the Non-Acoustic Underwater Imaging Branch of the Naval Coastal Systems Station, and have flown on STS-97 for the Structures Directorate of the NASA Johnson Space Center. While all of these sensors are capable of high resolution imagery, none of these sensors have the ability to identify objects, characterize objects, or report updated kinematic and dynamic data. This project will develop the ability to perform object tracking and characterization. The advantages of such a system are that it does not require cooperation from the target and it can operate at extended ranges. The functionality of such a system is twofold. First, it can be used to characterize and improve the deployment of known objects. Second, it can be utilized to characterize unknown objects and then categorize these objects into classes. Ultimate success will culminate in a final (staged) demonstration where data are acquired for multiple objects deployed simultaneously and the dynamics of these objects are determined in near real time.

## Accomplishments

One of the most significant successes developed when the team was performing a needs assessment of potential users. During this process, the team determined that future national security needs will require a fast, accurate tracking platform capable of supporting multiple sensors and a small, lightweight, deployable platform providing multiple views of the target scene. To date, the team demonstrated tracking targets, registering the images resulting in a 3-D point cloud from Laser Dynamic Range Imager (LDRI) data. The current algorithms automatically track features but require a user to select the search area. The next step, already under way, is to develop techniques to automatically segment and track multiple independent objects via model based algorithms. Our effort has been leveraged into additional work from NASA on the LDRI project and a Cooperative Research and Development Agreement (CRADA) with Boeing.

# Simulating Human Behavior for National Security

*71943*

*M. L. Bernard, M. R. Glickman, R. L. Waymire, P. R. Wolfenbarger, D. H. Hart, P. G. Xavier*

## Abstract

This effort is focusing on what we believe is a significant technical gap in existing modeling and simulation capabilities: the representation of human behaviors. In particular, we are developing models that will realistically represent intra- and intergroup interactions between virtual humans and human controlled avatars as they interact with their environment. We are conducting research to enhance our existing cognitive models to include sociologically plausible behaviors most important for representing human interactions. This work will integrate and leverage past achievements, including Umbra and the cognitive modeling framework, to build a modeling and simulation capability that will serve as the technical foundation for subsequent national security analyses and security personnel training.

Through this large scale development and integration effort, we intend to achieve a scalable capability that allows us to model large numbers of virtual humans and their interactions with high fidelity cognitive modeling of key personnel or smaller numbers of participants. Development of computationally efficient code will allow us to run many instantiations of our model in a Monte Carlo fashion and provide the resultant body of outputs to optimization algorithms enabling state-space searches in support of national security applications. We anticipate that the technical foundation developed by this project will serve as the basis for assessments of proposed security enhancements and/or new technologies that will aid in the generation of recommendations for security strategies. We envision that the capabilities generated by this project will serve as the technical foundation for next generation training tools for Department of Energy response forces and Department of Defense military personnel.

## Accomplishments

A major accomplishment was an initial demonstration featuring three interacting simulated humans whose high-level behaviors are driven by cognitive models. In the demonstration scenario, security forces clear an office environment of intruders. All of the simulated humans can sense each other, react to each other, and move about in the simulated environment. In this proof-of-concept demonstration, simple, semiplausible cognitive models, each running on an instance of the Sandia cognitive runtime engine with active memory (SCREAM), control the high-level behaviors of three intruders. A state machine technology controls the security force behavior.

While this demonstration showed preliminary implementation of perception control, it is further exemplified in a demonstration under development. The simulated humans perceive patterns of cues from the environment and respond appropriately to different environmental situations. If their environments change, so do their perceptions regarding that environment; consequently, their behaviors change as well. We developed a preliminary action generation, or motor-level cognition model, that transforms abstract actions generated by high-level cognition to actions for execution by a simulated physical human model. We exercised the first implementation of this behavioral layer in a demonstration. We developed a new state-machine based framework and are now using it to implement this layer in active work.

On a software engineering level, the entire project code base is modularized into a standard form that will enable other groups to start developing and using cognitive model-driven simulated humans in other Umbra-based projects. In addition, we developed a reactive control level for all characters and a joystick control interface for noncognitive characters.

We continue to focus on developing a method for combinatorial knowledge elicitation and demonstrating the ability for multiple simulated humans, of at least two different baseline types, to interact.

## Refereed

M.L. Bernard, P. Xavier, P. Wolfenbarger, D. Hart, R. Waymire, and M. Glickman, “Psychologically Plausible Cognitive Models for Simulating Interactive Human Behaviors,” presented and in *Proceedings of the Human Factors and Ergonomics Society*, Orlando, FL, September 2005, vol. 1, pp. 1205–1210.

M.L. Bernard, P. Xavier, P. Wolfenbarger, D. Hart, R. Waymire, and M. Glickman, "Modeling Psychologically Plausible Interactive Human Behavioral Models," presented and in *Proceedings of the 2005 Conference on Behavioral Representation*, Universal City, CA, May 2005, vol. 1, pp. 1500–1505.

## Other Communications

M.L. Bernard, P. Xavier, P. Wolfenbarger, D. Hart, R. Waymire, and M. Glickman, "Human Interaction Models for Training and Tactics Development," presented at the RedTeam 2005 Conference, Albuquerque, NM, 2005.

# Adaptive, Peircean Based Decision Aid

*79829*

*M. E. Senglaub, H. E. Link, M. A. Risenmay, E. M. Raybourn, M. L. Bernard*

## Abstract

The effort in this research and development activity focuses on a decision science problem that concerns adaptation and innovation. The human decision maker, as opposed to a machine, has an ability to solve original problems. Abduction, a hypothesis generating function, is the key Peircean component that enables rational logic-based reasoning and distinguishes it from less capable reactive technologies. Using the technologies associated with formal concept lattices and developing an analytical architecture based on Hawkins ideas of the neocortex, provides a framework for enabling the Peircean reasoning model to be invoked. This phase of the effort provides the basis for how we make decisions. There is a growing body of literature that is concerned with what we make decisions about and when we make them. This might be characterized as a form of metacognition. This area of reasoning research is cutting edge, and work in the literature may provide the theoretical underpinnings for engineering a solution to this aspect of reasoning. The improvised explosive device (IED) domain of application is co-evolutionary and requires a unique knowledge base growth characteristic to provide a theoretical foundation from which to generate hypotheses. We developed a model of process reasoning that builds on the power of formal concept analysis and melds nicely into the game theoretic infrastructure.

Instantiation of the Peircean model of reasoning will provide the capability for a machine to operate in unconventional warfare environments and to control highly complex massively integrated systems, and will provide a foundation for managing the explosion in information.

## Accomplishments

We designed the co-evolutionary game theoretic environment in which adversaries compete against each other. This construct required a process reasoning as well as a concept reasoning capability integral to the system. We developed the process reasoning engine based on a mathematical transformation between a formal concept lattice and a Markov transition matrix. That matrix provides the basis for the process reasoning engine. We identified a temporal formal concept analysis technology and are exploring it as an alternative to the Markovian approach.

We commenced initial translation of Mills canons into knowledge operators. These operators function similar to mutation and crossover operators in genetic algorithms, but in a knowledge space.

We completed the initial design of the Peircean reasoning engine. We must refine this design before coding can begin.

Research into formal modal logics provided a basis for developing a decision making paradigm for command and control. We are using this basis as the design reference model for the game engines and the design of the decision aid. This research also provided insights into information operations in general. We applied this research to information operations problems associated with vulnerability assessments for complex systems.

Work in the literature related to the structure of the neocortex provided a number of insights into design of the information systems and led us to develop new concepts of data and information fusion systems.

Finally, we conducted a review of Peirce's ideas on pragmatism to organize and focus some consultation needs.

## Refereed

M.E. Senglaub and D.L. Harris, "A Modified Perspective of Decision Support in C2," presented and in *Proceedings of the 10th ICCRTS*, McLean, VA, June 2005, CD-ROM.

# Towards a Stochastic Foundation for Information Operations

*79830*

*R. E. Carlson, S. Y. Goldsmith, C. A. Phillips*

## Abstract

A primary context for future information operations (IO) will be collections of intelligent, networked nodes that communicate with other nodes over terrain and static and dynamic infrastructure as dynamic teams. The cyber component of each node will incorporate a possibly decentralized controller that guides the behavior of each team member or infrastructure element to an overall optimal outcome. It is the hypothesis of this work that a stochastic representation of intelligent agent collectives will help to anticipate and measure IO effects, the causal chain that produces the effects, and optimal defensive response. The purpose of this research is to identify and begin to characterize such stochastic extensions for IO.

## Accomplishments

We investigated two directions: the optimal internal state of a cyber agent and the representation for optimal decision planning in stochastic environments. We were able to formulate the problems as a queuing model where service deteriorates over time. As service deteriorates, so does system performance, and, if left unchecked, the system dies. The system is managed by an agent that allocates resources to jobs and also maintains the system. We model deterioration as a shock process that is not directly observed by the agent. The agent, however, has the capacity to find and correct problems caused by the shocks.

We developed criteria for determining adequate service performance. One criterion is to keep the total amount of service allocated to jobs above a threshold determined by the traffic intensity. We point out that the traffic intensity is the random process the agent is controlling. Since the traffic intensity depends upon the shock process that is not directly observable, the traffic intensity is also not observable to the agent. One consequence of the intensity not being observable is that the transition function governing the system is also unknown.

Using ideas from nonlinear filtering, we construct the dynamics of the queuing system. This enables us to efficiently simulate the system using discrete event simulation methods. In addition, the filtering formula gives the conditional probability of the intensity given the current system state, usually the queue length. Thus, we are able to estimate the probability that the system is meeting the performance criteria.

Regarding representation of a multiagent system, we also identified that the fundamental problem of state-space explosion appears to be manageable through a factored partially observable Markov decision process (POMDP) representation that can be approximated by a linear combination of basis vectors, where the approximation can be exactly and compactly represented as a linear program.

There are two important consequences of this representation. The first is that we can achieve an exponential compaction of the state-space, allowing significantly larger POMDPs than previously thought possible. Secondly, it leverages the ongoing effort at Sandia to represent large scale linear programs in a parallel processing environment. The result is that it may be possible to produce realistic simulations of large scale cyber collectives in competition for resources and dominance.

# Design Tools for Complex Dynamic Security Systems

*79831*

*K. N. Groom, C. L. Lewis, B. R. Rohrer, J. J. Carlson, J. J. Harrington, R. J. Bickerstaff, R. H. Byrne, J. B. Rigdon*

## Abstract

The goal of this project is to develop modeling, simulation, and design 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 is often identified by the appearance of emergent or complex behavior from relatively simple rules. A complex dynamic security system takes advantage of these simple rules to exhibit the desired complex behavior. By combining complexity theory concepts with the dynamic security system concept, we hope to revolutionize the design and performance of security systems. We plan to investigate several technical areas over the course of the project. These areas include, but are not limited to: probabilistic algorithms, optimization algorithms, and intelligent learning algorithms. In addition, we plan to develop a dynamic security system testbed to validate the algorithms and tools developed under this effort. We will exploit synergies with other projects to enable the aggressive goals of developing a dynamic security system testbed in conjunction with the modeling and simulation tools. The security system testbed is critical to allow a spiral development process of iterative analysis, simulation, and experimentation. Although we will focus on the dynamic security application, our goal is that these tools will generalize to other classes of complex systems.

## Accomplishments

(1) Dynamic Security Testbed

We designed, built, and tested a dynamic security testbed consisting of a 100 node wireless sensor network (WSN), an unmanned ground vehicle (UGV), and an unmanned air vehicle (UAV). We are using this testbed to verify and validate the complexity theory and the computer simulations performed in this project.

We upgraded the AutoNav UGV from DOS to Linux. Leveraging the work of the team on LDRD Project 67037, “Enhanced Perception for Remote 3-D Mapping of Unknown Indoor and Outdoor Environments,” we added a laser scanner and an inertial measurement unit. The vehicle can now move autonomously in an unstructured outdoor environment. We are using this UGV to intercept intruders detected by the WSN.

The UAV can autonomously fly via global positioning system waypoints with a pilot utilized for landing and takeoff. Continuous video is streamed from a pan/tilt mounted camera. The UAV is being used to locate and track intruders that have been detected by the WSN.

(2) Complexity Theory Research

We investigated emergent behavior rules for the coordination of UAV and UGV collectives and achieved positive initial results. We explored percolation theory as a WSN communication analysis tool and investigated it as a tool to determine the necessary WSN density to detect an intruder. We expanded the partially observable Markov decision process for emergent behavior to allow power optimal tracking of multiple targets in a sensor network and ported it to UMBRA. We initiated research on entropy theory to determine its promise for emergent behavior. We began work to create a complexity roadmap and to plan a workshop with the goal of making Sandia a recognized leader in complex adaptive systems.

# Knowledge Discovery via Sensor Fusion in Structures and Ad Hoc Networks

*79832*

*T. J. Draelos, N. G. Brannon, G. N. Conrad, D. L. Harris*

## Abstract

Fixed and mobile platforms in civilian, military, or international monitoring share the potential to benefit from diverse information from various sources. Electronic sensors, intelligence and cyber reports, and local health and law enforcement bulletins all provide data that, in the aggregate, could significantly improve situation awareness. These modalities all generate large volumes of information that, if unfiltered, often overwhelm users. Local and regional processing algorithms, assuming limited computational power, are important to reduce data transmission, processing, and storage.

We propose to implement a sensor fusion capability in hardware to provide local, in-the-field fusion, and to design a computational engine for sensor fusion and situation awareness that integrates elements of supervised, unsupervised, and reinforcement learning. We have a state-of-the-art adaptive machine learning system design for sensor fusion and situation awareness and intend to advance it to a demonstrable human-in-the-loop decision support system for force protection. While this design is pushing the envelope of machine learning research, it matches the needs of situation awareness and human-in-the-loop decision support extremely well.

We will develop the capabilities to (1) accommodate diverse information sources as input, (2) generate situation awareness evidence, and (3) assess situation awareness based on that evidence. We will also develop a decision support user interface for force protection operators, tailor our system to a real-life force protection application, and then test and evaluate the performance of our system.

We are collaborating with the University of Missouri-Rolla, which has extensive experience in the targeted machine learning technology.

## Accomplishments

The team achieved accomplishments in three categories.

- *Sensor Data:*

(1) University of Wisconsin SensIT data: We acquired, reviewed, and performed limited testing on multisensor data from a Defense Advanced Research Projects Agency (DARPA) exercise for sensor fusion target detection and identification.

(2) Sandia protective force: We interacted with Sandia protective force personnel for the purposes of interviewing decision makers and acquiring sensor data.

(3) Coordination with virtual perimeter security (VPS): We had discussions regarding the sensor fusion needs of the VPS LDRD project.

(4) Exploration of state-of-the-art sensors: We held discussions and observed demonstrations of sensors by intrusion detection personnel.

(5) Water quality data: We performed analysis of water quality data supplied by the Sandia geohydrologists.

(6) Umbra: We explored/training in the use of Umbra for the purpose of simulating force protection scenarios and collecting realistic sensor data.

- *Smart Sensor:*

(1) Field programmable gate array (FPGA) implementation of sensor fusion: We completed initial implementation of the ARTMAP portion of our sensor fusion system on an FPGA.

(2) Neural network-based strain sensor: We began development of a strain sensor using simple physical sensors and a simple neural network.

- *Sensor Fusion and Situation Awareness/Decision Support:*

(1) Literature review: We completed an annotated sensor fusion, situation awareness, and decision support bibliography that guides our work.

(2) Computational engine for sensor fusion and situation awareness: We completed the design of our computational engine for sensor fusion and situation awareness that integrates elements of supervised, unsupervised, and reinforcement learning. The design pushes the envelope of machine learning research and matches extremely well the needs of situation awareness and human-in-the-loop decision support.

(3) Decision support GUI (graphical user interface) design: We completed the design of a meaningful and actionable operator interface to conform to the needs of force protection decision makers.

# UGS Concept and Technology Development for Enhancing Boost Phase Detection

*79836*

*A. B. Doser, P. D. O'Malley, J. L. Kalb, D. C. Craft, J. W. Giron, W. T. O'Rourke*

## Abstract

Unattended ground sensor (UGS) systems are a key technology in the areas of homeland defense, military operation, and commercial industry. Further development and integration of smart sensors, wireless networks, and real time algorithms will be essential for future UGS intelligence, surveillance, and reconnaissance (ISR) applications. Several ISR applications, such as national missile defense boost phase detection, require a unique sense, decide, act, and communicate architecture (SDAC). The SDAC architecture for these systems must accommodate two widely disparate operational scenarios and conditions. The two scenarios account for the two project objectives.

The first objective is to detect the boost phase with a high probability of detection while also having an exceptionally low probability of false alarms. This requires covert, low power, multimodal, smart sensors in a tiered classification scheme. This year, we developed algorithms to meet this target and the defined concept of operations (CONOPS).

We determined and reported network data rates and communication options. To accomplish the second objective of reporting, the system must quickly transition to a more capable sensing and processing state for target tracking and then queuing of the response assets. We will demonstrate much of this technology in FY 2006. In this operations condition, we placed a priority on gathering, communicating, and processing high fidelity information in real time. The project goals are development of a smart sensor architecture and CONOPS, research and development of low latency communications to enable high fidelity fusion, and research and development of real time software algorithms to locate and track targets. By the conclusion of the project, we will showcase the algorithms in a demonstration using actual sensor data.

## Accomplishments

The milestones for FY 2005 included: defining CONOPS, quantifying operational objectives, first order sensor selection, data collection, defining communications criteria, performing detection proof of concept, and completing sensor down selection. We defined CONOPS through meeting with knowledgeable parties and potential users. We further refined CONOPS through advanced literature searches. In addition, we established that a similar approach has never before been undertaken or reported in the open source literature. We demonstrated workstation based detection capability of an intercontinental ballistic missile (ICBM) using data collected from UGSs and using available launch data from previous tests. We developed a personal computer (PC) based beamforming algorithm for wide band sources; the source's direction of arrival can be determined. Using multiple arrays, we can estimate the location of the source. From localization, we can determine the direction of travel. The accuracy of the locale is directly dependent on the individual array apertures and the line-of-sight (LOS) difference of the arrays to the source. This assumes LOS of the source to the array is unobstructed.

We exploited wavelet techniques to develop a time/frequency based acoustic detection algorithm. This algorithm demonstrated zero false alarms for a library of signals. We demonstrated that geophone seismic data could be used to provide earlier event signature detection and event discrimination. We determined and reported network data rates and communication options. Due to a postponement in flight schedule, we were not able to perform a live data collection by the target date. We secured necessary equipment to proceed with data collection when the date arrives. Although we postponed the data collection, we were able to leverage existing data collected from previous tests to meet the remainder of our goals.

# Non-Inertial Electric Field Void Sensor

*79837*

*G. M. Loubriel, A. Mar, P. D. Coleman*

## Abstract

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. Unfortunately, the accelerometers are not very reliable and detonation may not occur at the optimum location. In this project, we investigated the feasibility of using alternative non-inertial sensors that do not rely on projectile acceleration to sense whether the projectile is in a void (air space), and that develop a history of the voids they encounter. This type of sensor provides a new paradigm to achieve fuzing for optimal effect. The sensors can be made small, and some of their subsystems have demonstrated high-g tolerance.

## Accomplishments

Experimental measurements indicate excellent target discrimination capability. We investigated concrete, sand, water, and black soil versus air. An issue with the sensors is whether the debris field ejected from the penetrator as it breaks through the material would affect the interpretation of the sensor's signals. To understand the nature of the debris field, we used a shock wave solid mechanics code (CTH) developed at Sandia. We modeled a 14 inch long penetrator passing through a 10 inch thick concrete wall as an example of a possible debris field. The penetrator outran the fragments; this was an encouraging result for interpretation of the void sensor return signals.

Examination of the data from earlier penetrator work confirmed the results of these analyses and measurements. The results also suggest a path forward to creating useful fuzing sensors that operate in minimal time and occupy a very small space. In total, the project indicates that the sensors could be a very valuable tool for optimizing the effectiveness of penetrating munitions.

# Large Area Metallic Photonic Lattices for Military Applications

*79838*

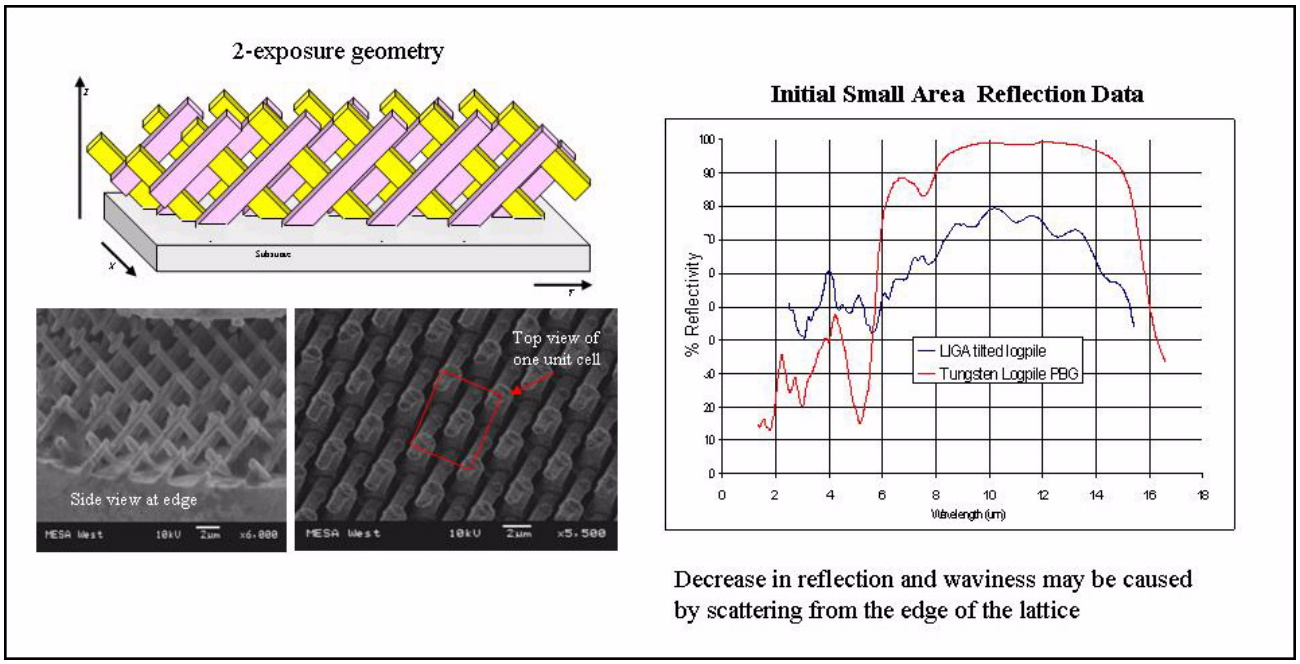
*F. B. McCormick, J. G. Fleming, S. H. Kravitz, D. W. Peters, W. C. Sweatt, W. G. Yelton, I. F. El-Kady, M. Watts, U. Krishnamoorthy, C. Schmidt, J. C. Verley, J. D. Williams*

## Abstract

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, display). However, current PBG fabrication techniques offer limited materials choices (tungsten, aluminum) and small areas (i.e., square inch rather than the square meter areas needed in these applications). We brought together Sandia expertise in PBG design/modeling to optimize PBG structures and to use new materials. We will also leverage 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 work is to demonstrate a 1m$^2$ sheet of three-dimensional (3-D) full bandgap photonic crystal. We demonstrated two new PBG material designs fabricated in gold using LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) and contact lithography processes.

## Accomplishments

We surveyed the theoretical design space while also experimentally exploring three specific fabrication approaches: a multilayer gold cubic array of cubes design (COAC), a holographically formed lattice plated in gold (in collaboration with Oxford University), and a LIGA-fabricated tilted logpile in electroplated gold. The theoretical work pursued both a general survey of PBG design forms and materials and a study of the manufacturability, tolerances, and trade-offs. We analyzed our current set of design software




*LIGA-fabricated tilted-logpine photonic crystal.*

tools and identified the trade-offs and desired development plans associated with each.

We developed multilayer lithography, alignment, and plating techniques for infrared (IR) wavelength PBGs that we can transition to nanoimprint lithography as a means to achieve visible wavelength PBGs. We developed two mask sets to fabricate a gold logpile design to compare to our tungsten logpile crystals and to fabricate a new COAC design.

The impact of various polymer filler materials is currently under modeling and experimental investigation. We investigated a variety of IR polymer filler materials (benzocyclobutene [BCB], several cycloolefins, polynorbenes, polyethelenes, and polymethylmethacrylate [PMMA]). We characterized the most promising ones using IR ellipsometry and Fourier transform infrared (FTIR) spectroscopy to provide complex refractive index data for the design models. We identified a low loss branched-polyethylene polymer with the appropriate solubility and processing attributes.

While the polymer investigation was under way, we completed preliminary fabrication of the COAC design using BCB as the filler polymer, and 2- and 3-layer structures are currently in testing. To investigate holographic PBG material fabrication approaches, we collaborated with Andrew J. Turberfield's group at Oxford University, obtained samples of their holographically exposed SU-8 molds, and gold plated them. Stress fracturing currently limits the sample sizes achievable with this approach to $< 1$ mm$^2$, and we developed a segmentation mask for them to help address this issue. We developed a two-exposure LIGA process to create a tilted logpile PBG structure. We performed experiments to minimize feature broadening effects, including the design of a 0.5 µm thick gold test mask and a 0.85 µm thick gold deep x-ray lithography (DXRL) mask. We used these masks to create 0.9 µm resist patterns exposed at a 45 degree angle into 18–25 µm of PMMA, into which gold was electrodeposited. Preliminary measurements show a moderate PBG similar to that of our tungsten logpile structures. We submitted five technical advances on high volume LIGA-based PBG manufacturing, novel magnetic materials, minimization of feature distortion during electroplating, and precision control of the complex dielectric constant in optical materials.

# Network and Adaptive System of Systems Analysis Methodology

*79839*

*D. J. Anderson, B. P. Van Leeuwen, J. E. Campbell, C. R. Lawton*

## Abstract

Considerable progress has been made in the development of system-of-systems (SoS) design and decision support methodologies for use in analyzing large scale, complex integrated systems such as the Army's Future Combat Systems (FCS). While providing substantial capabilities for evaluating a complex SoS, the current methodologies do not support critical issues associated with assessing SoS network-centric performance. Among these issues are adaptive communications and data networks, SoS-level effects of embedded diagnostics and prognostics, and optimization of SoS effectiveness. Networks and prognostics introduce dependencies and nonlinearities that can dramatically affect SoS performance. Treatment of networks and diagnostic/prognostic effects is crucial to realistic analysis of the FCS. This project will focus on advancing the current SoS assessment capabilities to address these critical needs. We will evaluate and develop network modeling approaches as needed. We will research and develop methods for adequately capturing the effects of embedded diagnostics and prognostics and the associated error rates, dependencies, and adaptability. We will evaluate and adapt current research in stochastic, multiobjective optimization techniques, as appropriate, for use in maximizing SoS effectiveness while minimizing logistics support and costs.

Successful completion of the proposed research will enable Sandia to maintain its leadership role in possessing unique capabilities and tools for analyzing complex SoS.

## Accomplishments

(1) We constructed an example network communications model that portrays a network of command and control vehicles.

(2) We collected data and information on network design, architecture, implementation, and specific FCS network information.

(3) We developed initial versions of network component state models.

(4) To account for dynamic and complex network modeling, we defined new concepts such as "super-system" that we will implement as an enhancement to the current SoS software.

(5) We conceptualized an approach for simulating the effects of prognostics and health maintenance (PHM) on system sustainability and the necessary logistics support.

(6) We developed methods for treating failure mode dependencies.

(7) We installed and executed the SoS simulation software on a multinode cluster as part of the implementation of an initial optimization approach to FCS.

(8) We have identified the following challenges of SoS modeling and analysis of complex, adaptive data and communications networks and prognostics:

- The problem scale grows exponentially in complexity when the interdependencies introduced by the data and communications network are taken into account.
- 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 is how to model and quantify the performance of the network when the network functions at a level above the individual systems upon which it depends.
- SoS networks are expected to be wireless, mobile, ad hoc networks (MANETs). The topologies of these networks are randomly dynamic and based on protocols yet to be defined.
- The network depends on the systems containing its nodes, and the performance of the systems themselves depends on the network performance, presenting the possibility of irresolvable circular references.

# A Pointable Platform for Acquisition, Sensing, and Tracking from a Target Booster Front Section

*79840*

*L. A. Jones, W. J. Zubrzycki, M. C. Wanke, M. Lee, R. J. Lawrence Jr, S. J. Greene*

## Abstract

Novel concepts were investigated for acquiring and tracking targets with a pointable platform on a target booster front section. These concepts have applicability to a variety of National Missile Defense (NMD) applications. Three thrusts were pursued in this project: development of a front-section sensor pointing platform concept, investigation of a novel sensor technology for use on this platform, and investigation of phenomenology that might be sensed during an NMD intercept.

In the first thrust, we developed a concept for a sensor pointing platform in the front section of a target booster. An inertially stable platform within the front section utilizes commands to the front-section attitude control system for coarse pointing and a two-axis gimbal for fine pointing.

In the second thrust, we began an assessment of the feasibility and component-level technology requirements for an exoatmospheric sensor system capable of performing target discrimination and post-impact target typing functions, using the terahertz (THz) portion of the electromagnetic spectrum. Tasks included demonstrating the capability to measure THz absorption/emission spectra from various molecules in vapor phase, measuring physical properties such as transmission spectra of several different fabric materials used in targets, and developing a quantitative signal link analysis to determine required component parameters and distance limits for THz sensing.

In the third thrust, we conducted a set of experiments to show proof-of-principle that hypervelocity impact flash is a feasible and credible tool for missile-defense-related kill assessment and target typing. Results included (1) the establishment of infrared spectroscopy as a diagnostic tool on hypervelocity gas guns; (2) impact-flash measurements both on gas guns and on Sandia's Z-Pinch machine; and (3) the identification, reproducibility, and temporal correlation of materials involved in the engagement. These experiments significantly expanded the impact flash materials database for use in the design of sensors on front-section pointable platforms.

## Accomplishments

(1) Sensor Pointing Platform

We developed a concept for a sensor pointing platform in the front section of a target booster. An inertially stable platform within the front section was the front section attitude control system for coarse pointing and a two-axis gimbal for fine pointing. A pointing camera on the platform is used for acquisition of targets and the subsequent pointing of a bore-sighted large optic sensor. The large optic sensor collects science sensor data on long range objects and transmits the data to ground stations. Data gathered by the sensor pointing platform may be used for target scene validation, intercept impact scoring, and post-intercept scene characterization.

(2) Terahertz Sensors

We began assessing the feasibility and component-level technology requirements for an exoatmospheric sensor system capable of performing target discrimination and post-impact target typing functions by using the THz portion of the electromagnetic spectrum. The first task was to demonstrate the capability to measure THz absorption/emission spectra from various molecules in the vapor phase. We demonstrated the ability to collect THz signatures of a number of different chemical vapors, such as carbon monoxide, methanol, and water vapor, with the same very sharp spectral linewidth resolution that such molecules would exhibit in an exoatmospheric situation.

We then used the same THz spectroscopy capability to measure physical properties such as transmission spectra of several different fabric materials used in targets. We found that fabrics containing no metal, and that are opaque in the visible, could have significant THz transmission. Metallized fabrics proved more difficult to characterize. Smooth and continuously metallized fabric gave no measurable transmission from microwave through THz. However, THz transmission could be measured when there were holes, cracks, creases, or other breaks in the metallization. We found THz transmission to depend strongly on the detailed nature of the breaks in metallization and how such breaks were formed. We began an effort to systemati-

cally and controllably study the effects of breaking the metallization on THz transmission through the fabric.

Finally, we began a quantitative signal link analysis to determine the necessary component parameters and distance limits for THz sensing. We found that narrow band heterodyne receivers offer the best, though not the only, chance of achieving good signal-to-noise levels across useful standoff distances.

(3) Impact Flash Phenomenology

We conducted a set of experiments to show proof-of-principle that hypervelocity impact flash is a feasible and credible tool for missile defense related kill assessment and target typing. Our efforts resulted in the following accomplishments:

(a) We established visible and infrared spectroscopy as a diagnostic tool on our hypervelocity gas guns.

(b) We obtained impact-flash measurements on both gas guns and on Sandia's Z-Pinch machine, using both basic and operational materials.

(c) We demonstrated with these measurements that (i) we can identify materials involved in the engagement; (ii) the spectral data are reproducible; and (iii) temporal analysis of the spectral data correlates with target configuration, e.g., for spaced targets.

Materials and samples examined to date included aluminum, titanium, indium, and silica phenolic, as well as multilayer and spaced targets, using these same materials. Our work significantly expanded the impact-flash materials database for use in the design of sensors on front section pointable platforms.

## Other Communications

R.J. Lawrence, W.D. Reinhart, L.C. Chhabildas, and T.F. Thornhill, "Hypervelocity Impact Flash At 6, 11, and 25 km/s," presented at the American Physical Society Topic Group of Shock Compression of Condensed Matter, Baltimore, MD, 2005.

# Detection of Contraband in Fuel Tanks

*86641*

*L. E. Larsen, P. E. Patterson, R. P. Toth*

## Abstract

The purpose of the project is to detect distributed false compartments in fuel tanks. We previously demonstrated methods for detection of contraband in a fuel tank subdivided into two compartments: one compartment for fuel and the other (normally much larger) compartment for contraband. A counter measure to that success is numerous small compartments distributed in the tank. The physical methods used previously are not feasible for the current problem. We explored and tested new methods to detect many small compartments distributed in various ways within an otherwise unaltered fuel tank.

## Accomplishments

We tested these new methods against two target classes: (1) randomly distributed compartments scattered across the bottom of the tank, and (2) arrays of such distributed compartments similar to eggs in a carton.

The methods used for these two target classes were similar insofar as stimulus-response paradigms were used. The two target classes differed, however, by arrangement of the transducers and in the signal processing methods used.

We were able to detect both classes of targets in the tank at about the same threshold. The results with randomly distributed targets were especially encouraging in that the response varied monotonically with the load.

# Static RF Cavity Experiment for Penetration Probe Communications

*90495*

*C. O. Landron, D. L. Faucett, R. A. Bates*

## Abstract

The effective implementation of a wireless telemetry system into an earth penetrating weapon depends significantly on our ability to predict the propagation characteristics of a particular frequency in a given penetration environment. It is highly unlikely that there is a single solution for all of the potential earth penetrator applications due to variables such as diameter of the penetration cavity, anticipated depth of penetration, depth of backfill on top of the penetrator, and soil type. Therefore, characterization of different frequencies operating within different pseudo-penetration cavities will provide valuable information for radio frequency (RF) telemetry designers who will help select the best frequency range to be used for a particular wireless telemetry system in a given penetrator environment. The primary goal of this project was to characterize the RF propagation characteristics of various frequencies within several different pseudo cavities. Characteristics of most importance were: attenuation as a function of spatial separation of the transmit and receive antennas, attenuation as a function of fill material between the transmit and receive antennas, and magnitude of the reflection coefficient for the transmit and receive antennas while in the pseudo-penetration cavity environment. We conducted field test experiments using different frequency bands inside various sized cavities and measured the RF propagation and antenna loading characteristics. Data generated from these experiments provide reference information for RF telemetry designers correlating transmission loss as a function of cavity diameter and distance for a given frequency, bulk soil attenuation for a given cavity diameter and depth for a given frequency, and antenna loading as a function of cavity diameter and soil backfill. We will use the data gathered during these experiments to refine the simulation models that predict the performance of future wireless telemetry systems incorporated into earth penetrator instrumentation systems.

## Accomplishments

We designed and constructed an RF static cavity testbed. The testbed is composed of a mound of soil approximately 8 feet in height, 25 feet long, and 20 feet wide. We laid four different diameter (4", 6", 12", and 16") 19+ foot long PVC pipes horizontally, approximately 4 feet up from the base, with 5 foot separation between tubes. This testbed is a semipermanent structure and can be used to facilitate RF propagation in cavity studies beyond this project.

We conducted forward transmission studies between transmit and receive patch antennas in a given cavity diameter at UHF-, L-, S-, C-, and X-band frequencies, in 4" and 16" diameter tubes. We collected data with and without backfill soil in the propagation path, providing information related to attenuation as a function of distance for each frequency within a given tube diameter. The data also provided information on limited bulk dry soil attenuation as a function of frequency within a given tube diameter. We dropped studies within the 6" and 12" diameter tubes due to problems encountered with some of the test equipment. The 4" and 16" diameter studies were the most relevant to ongoing penetrator studies because they closely resemble the penetration cavity diameters produced by the hard and deeply buried target probe and BLU-109 penetrators.

We obtained reflection coefficient magnitude measurements on the transmit and receive antennas while they were inserted into the PVC tubes. Data collected will provide RF telemetry designers with valuable information related to the effect that a given diameter penetration cavity has with respect to the loading of the antenna. This information will enable the RF telemetry designer to incorporate buffer substrates required for antennas used in penetration environments. This will help to minimize the loading effects on the antennas, which will reduce losses associated with antenna detuning.

The testing we conducted for this project provided valuable information related to the trade-offs that must be considered when attempting to incorporate a wireless link into a penetrator telemetry system. Our studies appear to confirm previous assumptions regarding the viewing of the penetration cavity as a lossy waveguide. Additionally, the waveguide effect is more pronounced with increasing frequency and tube diameter, and results in reduced RF signal attenuation as a function of

distance. The trade-off is that the higher frequencies experience greater signal attenuation when propagating through bulk soil than would be experienced with backfill into a penetration cavity.

Data collected during this project will be valuable in refining existing simulation models to better match empirical observations. The improved simulation models will provide more accurate predictive capabilities for RF propagation within a penetration cavity. This will be beneficial in defining future RF telemetry designs incorporated into earth penetrators.

# Human Performance Modeling of Soldiers for SoS Analytics

*90496*

*C. R. Lawton, J. E. Campbell, D. P. Miller*

## Abstract

Human performance modeling (HPM) is a significant challenge for future systems modeling and analysis initiatives. Military operations can have negative consequences for soldiers who are relied upon to perform critical tasks. Being wounded might limit activities; however, driving a vehicle might still be possible. Other factors that can degrade performance for tasks include fatigue, inadequate training, low experience, and a soldier's work/rest schedule. Fatigue, for example, can produce deficiencies in soldiers' performance that can result in inefficiencies and errors, thus impacting the probability of task and mission success. The type of task is important, especially in network-centric warfare, where information absorption and communication are critical.

HPMs are mathematical algorithms that accurately represent selected components of human behavior. We propose to develop HPMs, cast these models into a state modeling framework, and integrate them into our system-of-systems (SoS) analytics. These models must be able to address human behaviors that are specific to the tasks required within the mission or scenario being analyzed.

## Accomplishments

The goal of this project was to demonstrate the impact of soldier fatigue on the performance of a Future Combat Systems unit of action, using an SoS simulation-modeling framework that incorporates elements of the mathematical models to simulate the effects of sleep deprivation on humans. After performing a literature and research review of current fatigue modeling, we selected a model to form the basis for mathematical models to incorporate into the SoS modeling framework. We selected an approximation of the performance-decrement function characterized by the SAFTE fatigue model. In the SAFTE, performance linearly decreases over time at a rate of 20% per 18 hours of sleep deficit. We included the temporal multiplicative impacts of sleep deficit on soldier task performance in a small example simulation consisting of 40 systems and 116 soldiers by increasing the failure rate of crew members associated with platforms. (Note: Accommodating sufficient run-time, this analysis could easily be scaled up to an entire unit of action consisting of approximately 1500 systems and 3500 soldiers.) For demonstration purposes, we constructed three experiments:

(1) A baseline model, where soldiers do not experience sleep deprivation and soldiers fail at a constant rate.

(2) Experiment 1, where soldiers are not allowed to sleep until the 54th hour of the mission.

(3) Experiment 2, where soldiers are deprived of sleep for the entire mission (72 hours).

Each experiment consisted of a 72-hour mission and ran for 500 replications. As elements (soldiers as well as hardware components) fail, systems are not available, and a measurement of the systems' operational availability ($A_o$) can be computed. We computed the overall mean operational availability for each scenario after the 500 replications. The results were: 92.5% $A_o$ for the baseline, 92% $A_o$ for the scenario that allowed sleep at the 54th hour, and 90.5% $A_o$ for the scenario that deprived soldiers of sleep for the entire 72-hour mission. This results in a total difference of operational availability of nearly 2%. In addition, if infantry carrier vehicles (ICVs) are looked at in isolation, operational availability (instantaneous availability) at the end of each scenario was as follows: 93.2% $A_o$ for the baseline, 92.4% $A_o$ for the scenario that allowed sleep at the 54th hour, and 59.9% $A_o$ for the scenario that deprived soldiers of sleep for the entire 72-hour mission. That is, by the end of the mission ICVs were significantly affected by the lack of sleep of the crew, while sleep at the 54th hour allows for significant recovery. (Note: The baseline failure rates associated with soldiers were notional, so absolute impacts of sleep deprivation should not be concluded.)

The example analysis demonstrates very clearly that the state model object (SMO) simulation framework developed at Sandia can be augmented with mathematical models drawn from the soldier fatigue research base. It also shows that this augmented methodology can clearly quantify the impact of fatigue on the performance of an SoS.

One alternative to representing the soldier as an

element of a system is to represent the soldier as a system in and of itself. That is, the soldier would be modeled as an autonomous system that would have elements and functions. When simulating failure due to only fatigue, this level of flexibility is not necessary. However, if more sophisticated cognitive modeling of the soldier is necessary to model numerous human failure modes (e.g., lack of training, stress, injury) or represent more complex interdependencies between and among other systems, then modeling the soldier as a system will be required. Modeling the soldier as a system would also allow for more detailed treatment of sleep, activity, and sleep replenishment functions in the human system (e.g., individualized sleep schedules could be analyzed).

# Interstitial Monitoring Technologies

*90503*

*M. D. Torgerson, L. G. Pierson, M. J. Berg, A. Berry, K. O. Ramotowski*

## Abstract

Recently, researchers at Sandia conceived of the notion of an interstitial monitoring device (IMD) that leverages the independence, stability, and simplicity of the peripheral component interconnect (PCI) bus. There is a myriad of security questions that may be solved by this technology. The IMD concept is a significant step forward in the world of information assurance. If implemented, this technology could be one of Sandia's defining technologies.

Even though the IMD concept is simple, there are a number of technical challenges that must be worked out to make this new information assurance technology viable. Building hardware to interpret the data flows in real time will be a challenge. To be practical, an IMD device must conform to some reasonable form factor. Finally, a significant aspect of all monitoring technologies is the "vector set" to be monitored.

## Accomplishments

We accomplished our initial objective to investigate a capability set for an IMD device. In addition to this, we began initial prototyping and implementation.

We designed and then implemented a decoder for the low pin count (LPC) bus in VHDL (VHSIC hardware description language). This involved studying the "Intel Low Pin Count (LPC) Interface Specification" and then designing the state machines and required behavior to decode and perform a level of correctness verification on LPC bus traffic. We used a logic analyzer to capture short amounts of real world LPC traffic. We created a number of test waveforms based on the captured LPC traffic and on all the different categories of valid traffic from the LPC interface specification. We used these waveforms to test the LPC decoder in multiple hardware simulations. We made several revisions to the LPC decoder design to correct decoding errors and to improve the performance of the design. The decoder now successfully decodes all of the test waveforms used.

While not yet completed, we made progress on interfacing the LPC decoder to the Altera Nios II embedded processor so that additional custom hardware connected to the processor, and software running on the processor, can perform further analysis and characterization of the decoded bus traffic from the LPC decoder module.

We developed a set of Java classes that will evolve into a bus sniffer. The current implementation is network based; that is, the bus data will be encapsulated in a user datagram (UD) protocol packet by the IMD and sent over a network link to a personal computer (PC) monitor. There, the Java program will extract the bus data and control bits. It then displays them in a manner to enhance readability, understanding, and correlation by humans. We completed the internet protocol/UD protocol portion and initiated work on the user interface.

# An Exploration of the Potential of Nanorobotics

*90504*

*J. F. Jones, N. A. Weir, D. P. Sierra*

## Abstract

The purpose of this project is to explore the scientific basis of nanorobotics to understand its promise and ultimately which research directions would best support the Department of Energy and other national security program objectives. Sandia is uniquely suited to investigate the potential scientific significance of nanorobotics, given our leadership in robotics coupled with our growing understanding of nanotechnology. This project includes an extensive literature search to review ongoing nanorobotics research activities and understand their scientific plausibility and potential value. Because nanorobotics is a new field, there is a certain amount of speculative research occurring. Our intent is to determine which areas in nanorobotics will hold up to critical thinking and experimentation and hence hold the greatest promise for future research.

## Accomplishments

We performed an extensive review of the literature in the area of nanorobotics. The main goal of this effort was to research and identify advancements/accomplishments made in this relatively new and emerging field. As a result, we hope to determine what routes in the area of nanorobotics are scientifically plausible and technically useful so that Sandia can position itself to play a role in the future development of nanorobotic technology.

The birth of nanotechnology is often associated with the talk given by Nobel Prize winner Richard Feynman entitled “There’s Plenty of Room at the Bottom.” In this talk, Feynman discusses the possibilities (i.e., in principle) of what is now commonly referred to as nanotechnology and how its advancement could potentially generate an enormous number of technical applications. Nanotechnology comprises technological developments on the nanometer scale, usually on the order of 0.1 to 100 nm. Advances in the field of nanotechnology expanded the breadth of potential applications tremendously in recent years, and nanotechnology research and development areas have been growing rapidly throughout the world.

We discovered that a great deal of the research in the US comes under the purview of the National Nanotechnology Initiative (NNI) that provides a framework for government agencies to collaborate. Many government departments and agencies participate in the NNI, including the Department of Energy, the Department of Defense, and the Intelligence Community. The NNI focuses on nine technical challenge areas; one of these areas targets robotics. There are extensive recent publications relating to the field of nanorobotics. The bulk of these papers fall into one of the following categories:

- Atomic scale robots composed of individually arranged atoms and molecules.
- Atomically precise manufacturing that involves the pick and place arrangement of individual atoms to create designer molecules or devices.
- Biologically inspired nanorobotics that involves the study of nanoroboticlike behavior of agents found in nature, such as motor peptides.
- Microscale robots composed of nanoscale components.
- Manipulation of nanoscale objects using probes with very sharp tips (tip radius of a few nanometers).

Of these categories, atomic scale robotics and atomically precise manufacturing appear highly speculative. Although there have been some scientific accomplishments, much of the work to date is purely theoretical, and many consider it scientifically controversial. Biologically inspired nanorobotics is a fascinating field of study that will undoubtedly help unlock many of nature’s mysteries. However, biologically inspired nanorobotics is a field best suited for study by life scientists, chemists, and physicists. Although scientific progress is slow due to the extreme difficulty involved in fabricating individual components, microscale robots composed of nanoscale components are plausible because many examples exist in nature. The manipulation of nanoscale objects using probe microscopes (nanomanipulation) comprises the bulk of published experimental work relating to nanorobotics. Nanomanipulation is interesting because it provides a

mechanism to study the properties of nanomaterials and a process to fabricate prototype nanostructures.

Because nanomanipulation is an emerging technology with identifiable scientific value, it is likely that the application of robotic technology to this field would be a worthy contribution. Doing so would improve the efficiency of nanomanipulation and enable more rapid discoveries relating to nanotechnology. Given Sandia's existing technology base in nanomanipulation and our extensive knowledge in robotics, we are uniquely suited to contribute in this area. Two issues need to be more fully addressed for the application of robotic technology in the area of nanomanipulation to proceed. These issues are the development of appropriate feedback sensors to enable automatic control at the scale of interest, and the identification of an appropriate nanoassembly problem on which to focus.

# Extension of Interferometric Synthetic Aperture Radar to Multiple Phase-Centers

*90505*

*D. L. Bickel, J. M. Delaurentis*

## Abstract

This project allowed for a brief study of extending interferometric synthetic aperture radar (IFSAR) methods to the case of multiple phase-centers and multiple targets within a range-Doppler resolution cell. In particular, this project focused on the two-target case with three to four phase-centers. We examined various methods for attacking this problem, including the development of a unique new method for estimating two targets with three or more phase-centers. We obtained simulation results for one of the methods. We also briefly compared the multiple phase-center problem in IFSAR with the multiple phase-center problem in ground moving target indication (GMTI).

## Accomplishments

We presented a study and description of various approaches to the problem. We focused on applying a couple of these methods to the two-target problem of layover in IFSAR systems.

One of the methods, considered in detail, uses a combination of multiple signal classification (MUSIC) and least-squares amplitude estimation. The other method is a new method that follows from examining the two-target van Cittert-Zernike equations that describe correlation between the data observed by the antenna phase-centers.

We developed a simulation and documented results from the MUSIC/least-squares amplitude method. We compared the IFSAR layover problem with the endoclutter GMTI problem and showed that the GMTI problem is a subset of the IFSAR layover problem.

We also investigated the problem of determining the number of targets within a layover region. Since our focus was on the two-target problem, we proposed a new method for determining whether there are one or two targets present.

# MEMS Oscillators for RF Data Transmission During High-g Penetration Events

*90731*

*R. H. Olsson, B. Jokiel Jr., J. G. Fleming, M. S. Baker, B. H. Uhl*

## Abstract

This report describes the development of low-g sensitivity micromechanical oscillators. We undertook the effort because the stability of crystal oscillators is compromised in high-g environments; this leads to communications and processing errors in radios and digital signal processors. We obtained lower vibration sensitivity by replacing the crystal frequency reference with a microelectromechanical system (MEMS) resonator. We summarized the causes, effects, and metrics of oscillator vibration sensitivity. We used finite element modeling of micromechanical beam resonators to determine techniques for low-g sensitivity resonator design. We decided on a square extensional resonator with a simulated vibration sensitivity nearly 4 orders of magnitude lower than the best crystal oscillators. We detailed the fabrication and characterization of these resonators. We presented a micromechanical oscillator that uses the square extensional resonator as a frequency reference along with techniques for designing low phase noise MEMS oscillator sustaining circuitry. We demonstrated a printed circuit board (PCB) level frequency synthesizer and transmitter for up-converting the output of either a low-g sensitivity crystal oscillator or a micromechanical oscillator to 2.4 GHz. We used this circuit to characterize the output frequency deviation of both the MEMS and crystal oscillators under g-loads up to 75 g.

## Accomplishments

We quantified the mechanisms responsible for vibration sensitivity in capacitively transduced MEMS oscillators including spring stiffening and electrostatic spring softening. We used finite element modeling of beam resonators to develop design techniques for achieving low-g sensitivity resonators. We simulated the vibration sensitivity of 10 MHz beam resonators over different bias voltages, electrode to resonator gaps, electrode configurations, and gap control. In order to significantly improve the vibration sensitivity over that of the best crystal oscillators ($1x10^{-10}$/g), MEMS resonators should operate at high frequency (> 10 MHz). They should also have differential drive and sense electrodes with an electrode to resonator gap greater than 100 nm that can be matched within 1 nm. Using information derived from the 10 MHz polysilicon beam vibration sensitivity analysis, we designed a 60 MHz, low g-sensitivity, square extensional resonator. The innovative mechanical, anchor and lower electrode design allows the resonator to achieve a simulated vibration sensitivity of $1.7x10^{-14}$, four orders of magnitude lower than the best available crystal oscillators. We fabricated these resonators in an 8 mask surface micromachined polysilicon process. Characterized resonators have a measured Q > 18,000 and a motional impedance of 4.5 kΩ, with a bias voltage of 23 V under 40 mTorr of vacuum. We designed PCB level oscillator sustaining circuitry, realizing a MEMS high-g oscillator reference. We designed this PCB to measure the vibration sensitivity of either a crystal oscillator or the MEMS- based oscillator. We used the PCB to characterize the vibration sensitivity of two high-g crystal oscillators. Efforts are also under way to measure the vibration sensitivity of the MEMS based oscillators.

# Detection of Antennas

*90732*
*L. D. Bacon*

## Abstract

The detection of wireless operations in and around sensitive areas is a difficult and important problem. The ability to detect communications systems, even when unpowered, is a new capability in the areas of detection of cell phones and other communications devices in Department of Energy-owned limited areas. The detection of unpowered systems increases the level of security by allowing detection of communications systems before their use.

We investigated a proof-of-concept approach for the detection of both active and inactive communications devices by illuminating the device with low level, radio frequency (RF) energy. Transmitters and receivers re-radiate harmonics when they are illuminated within their operating band whether they are on or not. When illuminated out-of-band, the re-radiation should be much weaker. We measured the re-radiation of two different consumer communications devices that differed by an octave in operating frequency: 462 MHz and 915 MHz. We illuminated the devices at low field levels from approximately one-half of their operating frequency to well above their operating frequency band. We measured scattered responses at the fundamental (illuminating) frequency and at 2, 3, 4, and 5 times the fundamental frequency. Our goal was to determine the required illuminating power density, receiver sensitivity, transmitter spectral purity, and typical signatures.

## Accomplishments

The illuminating energy interacts with the antenna, the preselector filter, and with the input/output semiconductor devices in a receiver/transmitter. The preselector modifies the amplitude of the harmonics generated by the semiconductor devices in the receiver. This provides a signature that can be used to distinguish between communications devices and scattering by other objects in the environment. The transceiver system under test operated in the 915 MHz band. An enhanced response in this band clearly stands out from the background. We found that the re-radiated harmonics were significantly larger when the illumination was stepped through the operating band of the units than when it was outside the band. Re-radiated harmonics were only slightly lower when the device was powered off. This opens up the possibility of detecting the presence of even unpowered communications devices. An additional advantage of using higher harmonics is the larger gain and directivity that can be obtained at higher frequencies. This should allow higher precision in locating the communications device.

In summary, the concept appears to allow practical implementation. Reasonably high signal levels are required at the target; this in itself is useful in reducing false alarms from the environment outside the illuminating beam. The required high signal levels can be achieved at reasonable distances. Coherent receiver techniques can be used to achieve processing gain. Additional potential applications include remote-controlled improvised explosive device detection and detecting enemy operations at a distance.

# Threat Mitigation via Direct Drive Currents

*90733*

*J. A. Alexander, S. L. Shope, C. A. Moya, J. M. Villalva, P. H. Primm, L. M. Lucero, G. J. Denison, T. L. Smith, J. M. Usher*

## Abstract

The goal of this project was to test for the effects of direct drive currents on or near the trigger electronics of improvised explosive devices (IEDs) and on vehicles. We built a Marx generator in a mobile trailer with a long transmission line to remotely deliver kiloampere class currents directly to various devices under test (DUTs). The currents could either be coupled directly via a hard conductor or could be delivered via an arc channel. The environment created during the arc channel method more closely models the intended delivery method for some proposed new technologies.

We tested five different representative IED remote trigger devices for effect. Some of these devices cannot drive a detonator directly and require an external drive circuit to provide the necessary current. The external drive circuits we tested are also representative of actual devices. There were two different external drive circuits, one latching and one non-latching. We tested these external drive circuits with remote triggers attached, and separately. We also tested a Dodge Neon for effects via direct drive currents by receiving current via an arc channel to the center of the hood.

This project investigated the various effects seen during testing as well as the approximate threshold level required. The number of variables available for a possible test matrix is vast. The data set from this effort is therefore not comprehensive. Nevertheless, the results do provide important data for furthering the study and development of devices for mitigating threats via direct drive currents.

## Accomplishments

We tested five different remote operated commercial off-the-shelf devices for the effects of direct drive currents applied on or near the device. These devices are representative of actual devices known to be used on IEDs. Some of these devices cannot deliver the required current on their own, or they do not stay activated long enough to function a standard low voltage detonator. These devices are normally found to be coupled to an external drive circuit that supplies the needed current and pulse width. For trigger devices that function long enough to hold the external drive circuit on, the external circuit is a non-latching topology. For the trigger devices that do not function long enough, the external drive circuit is a latching topology. Some of the trigger devices can drive the detonator directly. Effects on these devices varied from causing the device or system to function, which would initiate a detonation, to disabling the receiver without causing the device to function.

We performed the vehicle test to determine at what levels direct drive current applied to the body of the car would kill the engine, and at what levels would components be damaged where the vehicle could not be restarted. The car tested was a 1995 Dodge Neon. The arc channel was directed at the approximate center of the hood with the car running. On all but one test, we applied a ground strap to the frame that ensured a return current path. On the one test that did not have a ground strap, we observed that a surface arc occurred to ground on one of the tires to provide the return path. The below threshold current where the car was not stopped varied from 6.0–10.6 kA. A current level of 9.3–14.9 kA caused the engine to stop, but the vehicle would restart. A current level of 12.9–25 kA caused the engine to stop, and the vehicle required repair to be restarted. The components damaged were the electromechanical computers, the crank sensor, the map sensor, and the camshaft sensor. The crank sensor was the common failure point in a series of three tests. We diagnosed the vehicle and repaired it the first two times. We simply diagnosed it the third time. There was some indication that some of the failures may have had a cumulative effect from previous shots. In some test cases, the car would restart after a test, but the car performance was degraded (rough idle). There are not enough data to quantify that effect.

Direct drive currents are an effective threat mitigation technology. Causing IEDs to remotely detonate on command and to stop vehicles is valuable. We generated a number of ideas on ways to deliver direct drive currents for various real world threats. A more extensive look at effects will be necessary to properly size the most effective pulser technology and perform integration testing with delivery technologies.

# Evaluation of a High Voltage Switch for Penetrator Fuzing

*90797*

*C. L. Smithpeter, N. T. Davie, R. McEntire*


## Abstract

The purpose of this project is to evaluate the shock resilience of a solid state high voltage (HV) switch to reduce reliability uncertainty in the Sandia Intelligent Penetrator (SIPE) fuze.

If successful, the test results will provide confidence in the current design and motivate additional development projects. Alternately, testing failures will firmly establish the need for alternate switch designs.

Solid state switches are desirable for their low cost, simplicity of their firing circuit, and theoretical reliability. However, sled track testing has shown that solid state HV switches can fail during impact due to shock induced transient voltages. The packaging of the metal-oxide semiconductor field-effect transistor (MOSFET) controlled thyristor (MCT) switch was recently updated to mitigate previous failures in impact testing. The updated MCT has been integrated and successfully shock tested at low levels, but testing at higher shock levels remains incomplete. Thus, the confidence in the MCT design is low until testing is completed at the high g levels.


## Accomplishments

Our objective was to evaluate the shock resilience of an HV switch in impact testing. If successful, the switch will provide a small and reliable technology for initiating penetrating weapons. The packaging of the MCT switch was recently updated to mitigate previous failures in impact testing. We impacted a fuze with the MCT switch six times on the horizontal actuator facility at shock levels up to 24,000 g. We conducted the tests by propelling the test item sled into a stationary sled (~5000 lb) that held material to shape the shock pulse. The initial tests were lower amplitude 10,000 g decelerations lasting up to a millisecond in the axial direction. Later tests ramped up to 24,000 g and half millisecond duration in lateral directions. We designed these tests to mimic previous impacts that have failed switches. The recorded data showed the switch held back the high voltage during all impacts and functioned when commanded. We observed a ringing on the high voltage monitor as evidence of shock response in the electronics, but it could not be isolated to one component. The results indicate that the MCT switch can be reliable in penetrator weapons, given appropriate packaging and selection of surrounding electronics.

# Characterization of Rubber as a Light, Effective, Convenient Material for Armoring Light Military Vehicles

*91663*

*D. S. Preece, J. H. Stofleth, W. V. Saul, V. S. Berg*

This report is for official use only.

# Science Based Engineering and Cognition for Network Analysis

*91905*

*J. C. Forsythe, N. M. Berry, J. D. Basilico, M. D. Peters*

## Abstract

The purpose of this project was to design a concept system to enable the understanding of large scale human behavior in response to events. The concept involves the utilization of analysis and simulation on available information from the media to provide an understanding of the impact of past, current, and potential future events. We outlined the criteria for this system by looking at potential data sources. Next, we created a general design to fulfill the criteria. Then, we outlined a proposed implementation using existing Sandia and commercial technologies. After describing what parts of the design could be based on the existing technologies, we outlined and investigated the gaps to achieve the full system concept. Finally, we outlined methods for providing verification and validation for the system and defined the additional steps necessary to take the system from a prototype to a deployable implementation.

## Accomplishments

The project team accomplished the objective of developing a system concept and design for a system to provide tools for the understanding of large-scale human behavior through analysis and simulation. We designed the system as a set of tools to (1) help planners, strategists, and policy makers understand relationships between past and current events, and (2) to simulate responses to novel events that are given by the user. The proposed system will make use of Sandia's capabilities in modeling the cognitive, social, and economic aspects of human behavior in combination with capabilities in high performance computing.

The system is composed of three main parts: automated data collection, analysis, and simulation. We developed a set of criteria for each of the main components of the system to provide an outline for the behavior needed by the system. Since the abilities of the system are dependent on the data that are available, we analyzed potential input sources to the system in terms of difficulty of access, difficulty of extracting textual content, and difficulty of extracting the additional attributes needed by the system. The conclusion was that the easiest and most available media data to use for the system is web-based media such as online news, discussion boards, and personal web logs (blogs). More traditional media sources such as print, radio, or television would require overcoming large technical hurdles to extract the content necessary for the system.

Based on the established criteria and the available data sources, we developed the design of the system in the form of three tools that each target one of the criteria. The harvesting tool provides the automated data collection, and the extraction of the structured data needed by the other tools from the raw unstructured data. The analysis tool provides methods to inspect the data from the harvester and provides automated pattern analysis for discovering semantic, spatial, and/or temporal trends in the data. The simulation tool uses results from the pattern analysis of the analysis tool to create models that can respond to new events entering the system by simulating the cognitive and social aspects of human behavior.

After we completed the high-level design of the system, we detailed the recommended implementation. This included both the recommended existing Sandia technologies that could be leveraged along with commercial components that could be used in their place. The main technologies that make up the system are the cognitive model, Seldon, and N-ABLE. We defined the interfaces that these components would use to communicate with one another. We evaluated the existing technologies. We assessed the remaining gaps that must be filled and proposed methods for filling those gaps. The main gaps that we discovered were: joint analysis algorithms for finding patterns in the semantic, spatial, and temporal aspects of the data simultaneously; high performance implementations of the analysis; a population generator to create cognitive models for the simulation; a social network generator to create the existing social network that binds the models together; a simulation of information transactions for both cognitive models and the media; lightweight cognitive models; and a high performance simulation for both the information transactions and social networks.

Since the system concept involves looking at human behavior, we outlined methods to provide veri-

fication and validation of each component of the system. Finally, since the concept is for a prototype of the system, we investigated additional issues in taking the prototype to a deployable system such as languages, security, and automated source discovery.

# Smart Strike Weapon (SSW)

*93025*

*W. T. Gutierrez, M. L. Yee*

## Abstract

This project addresses the potential for a new generation of smart strike weapons (SSWs) designed to take advantage of additional intelligence, surveillance, and reconnaissance (ISR) information from ground sensors emplaced near the strike area. After an initial strike, the sensors may return information such as damage assessment, strike location, depth of penetration, and geologic constituency. The questions to be addressed are: How could this information be used to optimize a follow-on strike? What design options are possible to enable a weapon to adapt to perform an optimized strike? The purpose of the project is to explore answers to these questions and establish a foundation for eventually developing engineering and operational requirements for a smart strike weapon that leverages ground sensor ISR. This will hopefully lead to a new generation of adaptable weapons that work as an integrated system-of-systems together with ground sensors and other ISR assets.

## Accomplishments

This project examines the nature of the relationship between sensor information and the characteristics of SSWs to lay a foundation for the next generation of SSWs that work in a system-of-systems framework with real-time sensor systems. Using input from experts in various areas, including weapon guidance, fuzing, and detonation, and networked sensing and algorithms, we completed an analysis of the attributes and requirements of SSWs enabled by sensor information. We documented the results in a skeleton operational requirements document (ORD) for an SSW that leverages sensor ISR and a proposed roadmap for the development of future SSWs and sensor systems. This approach is different in that the SSWs and sensors are examined jointly in light of a system-of-systems approach instead of as distinct systems.

An SSW has a capability for adaptability and/or controllability of some kind. The goal is to use sensor information to direct the adaptation and/or control of the weapon to increase the probability of a successful strike. The principal areas of weapon adaptability and/or control are:

- Guidance
- Depth of penetration
- Arming, fuzing, and firing
- Detonation

The project results detail how sensor ISR can be used to benefit each of these areas.

# Parasitic Power Generation in a MEMS Device

*93390*

*D. K. Kholwadwala, B. R. Rohrer, J. W. Wheeler*

## Abstract

Small scale power supplies (microwatt to milliwatt range) are needed to make integrated, self-sufficient microsystems feasible. While some work has been done in this area, very few ready-to-use solutions exist. In this project, we defined specifications for a new small scale power supply for implantation in the human body. We surveyed currently available and developing technologies for candidate solutions. As a result of our survey, we determined that three technologies could potentially meet our specifications: batteries, thermoelectric generation, and mechanical energy harvesting. We then outlined solutions employing each of these technologies and described the steps necessary to mature the designs into devices suitable for testing.

## Accomplishments

We conducted a survey of power solutions for long term, small scale power sources that could potentially be implanted in the human body as in a medical device.

- Vibration harvesting:

Although lower than thermoelectric, vibration harvesting also has a sufficiently high power density to operate an implantable device. The mechanical nature of power production is particularly benign and poses no foreseeable difficulties with implantation.

- Thermoelectric generation:

The theoretical power density of a thermoelectric generator (TEG) is sufficient to operate an implantable device. It poses no obvious barrier to implantation.

- Subcutaneous photovoltaics:

With a theoretical power density on the lower limit of feasibility, the simplicity of this solution and the maturity of photovoltaic (PV) technology were factors in its selection. As with thermoelectric and vibration solutions, unlimited energy density, hence unlimited lifetime, was also a desirable feature.

- Conventional fuel cells:

Although the power and energy density of fuel cells are adequate, gases are typically required as fuel and/or given off as exhaust, making fuel cells poorly suited for implantation.

- Biofuel cells:

While their operation has been demonstrated in vitro, current biofuel cell technology has a very limited lifespan. There is no obvious solution to this problem; but if this issue is addressed, then biofuel cells should be revisited.

- Flywheels:

While flywheels may have adequate power and energy density for some implantable monitors, there remain two concerns significant enough to dismiss flywheels as a viable option. First, long term energy storage in the form of mechanical kinetic energy is likely to be compromised by friction. Second, any attempt to rotate the flywheel along any axis other than its own will produce significant procession torques, possibly causing the implanted device to wrench free of its placement.

- Radioactive sources:

Some thermoelectric and thermionic power solutions employ a sample of radioactive material as a source of heat or ions. Radioisotope thermoelectric generators (RTGs) have even been successfully employed in implantable devices. Beginning in 1970, mini-RTGs were implanted in human subjects as power supplies for pacemakers. A 1978 study tracking the first 1400 of these implants showed no electrical or containment failures. Eventually, more than 3000 mini-RTGs were implanted, and, as of 2003, roughly 150 units were still implanted and functioning normally. However, regulatory issues currently make the implantation of new radioisotope-powered devices unlikely for the foreseeable future. Since our goal is to identify technologies that can provide the next 10 years of implantable devices with adequate power, any power solution involving radioactive material was not considered.

# Hypersonic Submunition Dispense

*94019*

*J. C. Hogan, B. J. Ash, W. R. Engleman, M. E. Kipp, M. W. Pilcher, D. F. Teter, P. N. Demmie, J. Smith, J. H. Stofleth, W. V. Saul*

## Abstract

The goal of this project is to investigate the technical feasibility of dispensing conventional munitions from a hypersonic re-entry system. This research builds on modeling and experimental work done in the early 1990s where tungsten scored rods were explosively driven out of a sled track vehicle. We developed a new computer model of a re-entry body and used it to explore the requirement space. This phase of the work looked at submunition alternatives, including rods, small penetrators, and spheres, against real type targets. We studied submunition distribution diameter, mass, number, height of release, lateral explosive energy, and lethality against targets for each type of submunition, thus defining design goals. We used probability of kill as a measure of effectiveness. We performed a complementary analysis of the issues related to the release of submunitions at these Mach numbers. The primary focus was on tungsten projectiles as compared to steel. We chose the experimental matrix of projectile impact velocities and masses to bound the expected kinetic energy release conditions defined by the requirements model. In a parallel effort, we determined the potential weights and mass balance properties for the three types of submunitions and laid them out in actual re-entry bodies for the three types of submunitions, thus bounding the number that could be carried assuming a linear shape charge release. With the above information, we used an actual Department of Defense (DoD) re-entry body for experimental tests to validate the engineering methods. We developed computer models and material models for submunition deployment. We developed a plan for tracking a complex failure method and a complex flow method for an explosive tungsten foam system. We explored a workable system, in addition to explosive submunition deployment.

## Accomplishments

The accomplishments for this research are divided into the four research tasks.

Task 1 accomplishments are twofold. We considerably refined a submunition model developed at Sandia to better represent high Mach number release. We used the model to determine optimum delivery conditions for three different submunition types. We determined the delivery parameters from re-entry body velocity, angle of incidence, and accuracy plots. We used the results of the model and the trades to determine the system's requirements. We determined the engineering parameters of accuracy, target location error, submunition release height, size of cloud, velocity, drag of the submunitions, and lethality of the three submunitions against a representative Strategic Command (STRATCOM) target set. We used all these parameters to determine the probability of kill of the target. Multiple missiles were often required to achieve the desired damage. We performed aero-thermal heating from different release altitudes for the three submunition candidates. We studied the reaction of tungsten in the high heating environment and studied the chemical oxidation reaction with the atmosphere. The results indicate that additional studies will be necessary if a high altitude release is necessary. We consulted with and presented the results of the computer modeling to a panel of lethality experts for the Joint Munitions Effectiveness Manuals. The panel agreed with the methodology used for the probability of kill of the targets selected.

In Task 2, we investigated concepts for deployment of the submunitions, including linear shape charge and explosive expulsion, directly through the aero shell. We developed the initial phase of a computer code for submunition deployment and a materials model. We researched and applied the data from the test of cylinder submunition done in the mid 1990s to smaller higher velocity aero shells. We developed a process to acquire and implement approved materials into a submunition deployment system. Although we used the segmented tungsten rod as the baseline, we investigated other submunition concepts, including spheres and kinetic penetrators, for developing methods to deploy from an aero shell. In conjunction with the mechanical design effort, we developed a parallel processing capability for design and testing to perform future work. We performed explosive tests to validate the engineering methods and concepts considered.

In Task 3, we used a shock physics code to calculate the impacts of the three projectiles at normal incidence against two types of targets. We determined the residual velocity of each kind of projectile. We fed the results of these studies back into the operations analysis model to help determine the trade between numbers of projectiles, mass, and the resulting lethality. Higher numbers of projectiles increase the probability of hit, but the projectiles with greater mass have a higher lethality. We compared and contrasted tungsten and steel projectiles to help determine lethality. We then used these data in the operations analysis model.

We performed mechanical trade studies in Task 4 that defined the properties of mass and center of gravity for the different submunitions. We completed detailed two- and three-dimensional drawings with submunitions held in foam, tubes, and clamps.

# Nonproliferation and Assessments

## Development of a High Throughput Microfluidic Integrated Microarray for the Detection of Chimeric Bioweapons

*52706*

*J. A. West, B. J. Kirby, K. W. Hukari, R. Devay, G. A. Hux, B. Satterfield, T. J. Shepodd*

### Abstract

Genomic analysis tools have recently become an indispensable tool for the evaluation of gene expression in a variety of experiment protocols. Two of the main drawbacks to this technology are the labor and time intensive process for sample preparation, and the relatively long times required for target/probe hybridization. In order to overcome these two technological barriers, we developed a microfluidic chip to perform on chip sample purification and labeling, integrated with a high density gene array. We performed sample purification using a porous polymer monolithic material functionalized with an oligo(dT) nucleotide sequence for the isolation of high purity messenger ribonucleic acid (mRNA). These purified mRNAs can then be rapidly labeled using a covalent fluorescent molecule, which forms a selective covalent bond at the N7 position of guanine residues. These labeled mRNAs can then be released from the polymer monolith to allow for direct hybridization with oligonucleotide probes deposited in the microfluidic channel. To allow for rapid target/probe hybridization, we printed high density microarrays in the microchannels. The channels can accommodate array densities as high as 4000 probes. When oligonucleotide deposition is complete, we seal these channels using a polymer film, which forms a pressure tight seal to allow sample reagent flow to the arrayed probes. This process will allow for a real time target to probe hybridization monitoring using a top mounted charge-coupled device (CCD) fiber bundle combination. Using this process, we performed a multistep sample preparation to labeled target/probe hybridization in less than 30 minutes. These results demonstrate the capability to perform rapid genomic screening on a high density microfluidic microarray of oligonucleotides.

### Accomplishments

(1) We demonstrated rapid trapping (< 2 sec), preconcentration, and release of mRNA on porous polymer monolith materials.

This task focused on the development of prehybridization mRNA purification. We developed the ability to post-functionalize porous polymer monoliths with oligonucleotide sequences in order for use as mRNA purification devices. Having developed this ability, we then optimized protocols for the trapping and purification of rarified target analytes in a complex sample containing both deoxyribonucleic acid (DNA) and proteins. We realized the ability to perform this purification with unprecedented speed, less than two seconds.

(2) We developed a polymer film system for sealing open channel microfluidic devices to allow for DNA deposition on microfluidic microarray chips.

We previously developed microfluidic microarray chips, which offered the ability to perform post-chip fabrication DNA deposition using a robotic spotter. Previously, use of the chips required us to use a compression based system to seal the chips with a polymer

film. In order to perform real time detection using the fabricated and spotted chips, we developed a chemical process to allow for covalent attachment of a polymer film on the surface of these chips. We successfully developed a protocol using polydimethylsiloxane films functionalized with aminotripropylsiloxane to form a pressure tight seal of the open microfluidic channels. This seal allowed discrete fluid flow through the microfluidic gene array channels and enabled us to characterize the kinetics of the hybridization of the target and probes utilized on these chips.

(3) We demonstrated rapid gene array hybridizations using microfluidic microarray devices.

Using the newly developed process for sealing the microfluidic microarray chips, we then characterized the reaction rates of the RNA hybridization on the oligonucleotide spotted chips. In contrast to the standard protocols, which require up to 24 hours to perform microarray hybridizations, we realized the ability to perform RNA-DNA hybridizations in a time frame of 5 minutes or less using the microfluidic microarray chips.

(4) We collaborated with the U.S. Army Medical Research Institute of Infectious Diseases (USAMRIID) to test probe sequences and developed a pathogen virulence database.

We began developing a database for the detection of virulence factors from pathogenic viruses and bacteria. We successfully completed the first version of this query based database. Due to the pathogenic and toxic nature of these organisms, we do not have the capability to test the developed oligonucleotide sequences versus live agents at Sandia. To perform testing of this database, we collaborated with the USAMRIID to test a subset of these developed sequences. The testing of this database is ongoing.

(5) We developed a miniaturized integrated hybridization and detection platform for microfluidic microarray devices.

We realized that the intended use of these devices would require the development of an integrated hybridization and detection platform. We designed, fabricated, and tested the first prototype of this system. The platform includes both hardware and software components that enable the integrated use of the microfluidic microarray chips. The prototype system is designed to provide microfluidic and thermodynamic control, data acquisition, and analysis of the sample purification process, as well as the RNA-DNA hybridization reactions.

## Other Communications

J.A.A. West, K.W. Hukari, T. Shepodd, and G.A. Hux, “A High Density Microfluidic Microarray Platform for Rapid Genomic Profiling,” presented and in *Proceedings of the 8th International Conference on Miniaturized Systems for Chemistry and Life Sciences, μTAS*, Malmo, Sweden, September 2005, vol. 296, pp. 656–658.

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# Detailed Modeling and Simulation of Contaminant Transport in Architectural Spaces

*52711*

*C. E. Hickox Jr., B. M. Rush, J. S. Grieco, G. F. Homicz, D. K. Gartling, M. J. Martinez, W. S. Winters Jr., G. H. Evans*

## Abstract

This project focused on the development and application of modeling and simulation approaches for the prediction of contaminant transport in interior architectural spaces. The term "architectural spaces" includes the entire variety of enclosed interior spaces associated with various types of buildings, hotels, shopping malls, stadiums, airport concourses, seagoing vessels, and aircraft. Although the project is primarily motivated by the desire to reduce the risks from, and mitigate the effects of, attacks with chemical and biological (chem/bio) agents, the capabilities developed have direct application to the transport of contaminants associated with fires and other catastrophic events. These capabilities also have application to more conventional concerns associated with indoor air quality within interior spaces.

The goals of the project were: (1) to develop and implement a suite of validated computational tools that describe contaminant transport with sufficient accuracy to support credible risk assessments and consequence analyses, (2) to utilize the computational procedures to identify effective strategies to protect facilities and to mitigate the effects of attacks through the implementation of passive and active control strategies, and (3) to validate the simulation strategies through comparisons with existing data. Although not included as a specific goal, it is clear that the results of these investigations also have application to the effective placement and interpretation of data from chem/bio sensors. The computational approaches developed and/or investigated include (1) multizone models for highly simplified building geometries, (2) computational fluid mechanics (CFD) for detailed transport studies, and (3) linking of CFD and multizone simulations.

## Accomplishments

We developed new multizone simulation software and incorporated it into a software package called NETFLOW. The inclusion of compressible and nonisothermal effects in this package is an improvement over existing multizone simulation approaches; this enhanced capability is now used to enhance ongoing studies at Sandia. We used CFD simulations to investigate thermal effects on flows in rooms and thus aided the development of improved modeling strategies for nonisothermal flows in multizone simulations. We compared predictions made with NETFLOW with experimental data.

We used CFD simulations to investigate two- and three-dimensional velocity distributions, contaminant transport, and effects of density variations in several model room geometries. We employed a variety of CFD software packages in these investigations. These include: CURRENT, an in-house Sandia code for the simulation of two-dimensional fluid flow and transport; FUEGO, Salsa, KACHINA, and RIO, all of which are Sandia codes for the simulation of three-dimensional fluid flow and transport; and FLOW-3D, which is a commercial code for the simulation of three-dimensional fluid flow and transport. For certain specific geometries for which experimental data are available, we performed comparisons with numerical simulations. We performed limited scope, three-dimensional simulations with the CFD code FDS and visualization package Smokeview, which are available from the National Institute of Standards and Technology. We compared predictions from the FDS code with results of an initial experimental investigation of ambient velocity fluctuations in a room. We completed investigations into the linking of multizone and CFD simulations. The linking of these computational approaches offers the advantage of combining detailed CFD simulations in selected spaces of crucial interest with multizone simulations for the remainder of a complex interior envelope, thus providing a more efficient simulation strategy.

Based on the results of multizone and CFD investigations of contaminant transport, we identified effective approaches for the prediction of contaminant transport. In addition, the results indicate a crucial need for additional experimental investigations to improve modeling and simulation for contaminant transport in interior spaces.

## Refereed

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## Other Communications

W.S. Winters and B.M. Rush, "Application of NETFLOW to the 911-Bio Consequence Management ACTD German Village Exercise," Sandia Report SAND2004-3099, Livermore, CA, 2004.

# Ultrahigh Temperature Ceramics for Hypersonic Vehicle Applications

*52717*

*R. E. Loehman, D. W. Kuntz, P. G. Kotula, T. J. Boyle*

## Abstract

$HfB_2$ and $ZrB_2$ are potential thermal protection materials because of favorable thermal stability, mechanical properties, and oxidation resistance. The pure diborides are difficult to densify, and most bulk material is made by high temperature hot pressing with additives such as SiC. The overall goal of this project was to gain an understanding of the performance limiting features of ultrahigh temperature ceramics (UHTCs) based on the diborides $ZrB_2$ and $HfB_2$, and to use that understanding to make better thermal protection materials.

We made diboride ceramics containing 2 to 20% SiC by hot pressing and pressureless sintering at temperatures to 2200 °C. The relative densities of the pressureless sintered ceramics were all below 75% of theoretical, whereas the hot pressed ceramic densities were all greater than 95%. Dense hot pressed diborides with such low additive contents were not reported prior to our work. We studied bulk Hf and Zr diborides to determine their densification mechanism(s) with the goal of learning to make strong, pore-free ceramics by pressureless sintering techniques. High-resolution transmission electron microscopy (TEM) showed very thin grain boundary phases that suggest the presence of a liquid phase in sintering, but it is not clear that liquid phase diffusion controls the sintering rate.

We developed liquid precursors to Zr and Hf diboride coatings. The precursors chosen include derivatives of $M(BH_4)_3$ (M = Zr or Hf) because the binary tetrahydroborates are highly unstable species that decompose below room temperature. We prepared the 1,2-dimethoxyethane derivative, $M(BH_4)_3$(dme), by reacting $MCl_4$ with $NaBH_4$ in 1,2-dimethoxyethane (dme) at room temperature. We further used the dme derivatives to make diboride films and nanoparticles by MOCVD (metal-organic chemical vapor deposition). We deposited the films on graphite or infiltrated the precursors into carbon-carbon (C-C) composites. We determined decomposition by thermogravimetric analysis (TGA), structures and compositions by x-ray diffraction and analytical TEM, and oxidation by TGA.

We measured 4-point flexure strengths of hot pressed Zr and Hf diborides in air at 25 °C, 900 °C, and 1400 °C. The best specimens had strengths above 450 MPa at 25 °C with little decrease at 900 °C. The $HfB_2$-15% SiC composition had an excellent strength of 409 MPa at 1400 °C.

## Accomplishments

The specific objectives of this project were to:

- develop improved processing for $HfB_2$ and $ZrB_2$ based UHTCs;
- develop a database of reliable UHTC physical and mechanical properties;
- determine the relation between UHTC microstructure and properties using advanced electron microscopy analysis;
- determine the origin and type of failure for UHTCs subjected to realistic stresses and feed back that information to improve processing;
- develop liquid precursors to $ZrB_2$ and $HfB_2$ so the diborides can be applied as coatings to improve the oxidation resistance of other materials, for example C-C composites.

We substantially met or exceeded all of these objectives over the duration of the project. This year we concentrated on (1) completing our measurements of the thermal diffusivities of UHTCs, (2) measuring the high temperature strengths of bulk UHTCs, and (3) developing oxidation resistant coatings based on $ZrB_2$ and $HfB_2$.

At the University of New Mexico, H.P. Dumm determined the thermal diffusivities, specific heats, and thermal expansions of $ZrB_2$-SiC and $HfB_2$-SiC ceramics to 2000 °C, and correlated his results with sample compositions and microstructures. He found the thermal diffusivities and conductivities to decrease with temperature, SiC content, and porosity. Rule-of-mixture models suggested radiation across pores and lower than expected silicon carbide contents. The results provide a reliable, comprehensive assessment of the relation between UHTC compositions, microstructures, and high temperature thermal properties that can be used to design specific thermal protection schemes.

We measured the 4-point flexure strengths of 3 x 4 x 45 mm bars of hot pressed $ZrB_2$-SiC and $HfB_2$-SiC UHTCs at room temperature (RT), 900 °C, and 1400 °C. Average RT strengths varied from 200 to 500 MPa.

Initial examination of fracture surfaces suggested that the lower strengths were due to processing defects such as agglomerates and microstructural inhomogeneities. Three compositions had average RT strengths above 450 MPa. We judged the higher values to be more representative of intrinsic UHTC strengths and, therefore, further tested those compositions at 900 °C and 1400 °C. The results were:

$ZrB_2$-2.1% SiC: 500 MPa (RT), 425 MPa (900 °C), 200 MPa (1400 °C);

$ZrB_2$-20% SiC: 450 MPa (RT), 420 MPa (900 °C), 225 MPa (1400 °C);

$HfB_2$-15% SiC: 460 MPa (RT), 460 MPa (900 °C), 425 MPa (1400 °C).

The exceptionally high 900 °C and 1400 °C strengths for the $HfB_2$-15% SiC composition are very encouraging and indicate that the material is free of deleterious grain boundary phases that frequently limit high temperature strengths of ceramics.

We developed precursors to $ZrB_2$ and $HfB_2$ that can be applied to substrates as liquids and then heated to convert them to ceramic coatings. The precursors were derivatives of $M(BH_4)_3$ (M = Zr or Hf) because the binary tetrahydroborates are highly unstable species that decompose below room temperature. We prepared the dme derivative, $M(BH_4)_3$(dme), by reacting $MCl_4$ with $NaBH_4$ in dme at room temperature. We deposited the precursors on graphite or infiltrated them into C-C composites, which other than their susceptibility to oxidation have excellent high temperature properties. The dme derivatives were further used to make diboride films and nanoparticles by MOCVD. We used standard materials science techniques to determine optimum processing temperatures and structures and compositions of the resulting films. We determined the oxidation resistance of coated C-C composites in laboratory furnaces to 1700 °C and above 1800 °C in the National Fast Flux Solar Test Facility at Sandia. The UHTC films performed well in the tests and protected the carbon substrates as long as the films were pinhole free.

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# Ion Mobility Spectrometer–Mass Spectrometer

*52724*

*D. E. Austin, E. S. Varley, M. C. Lenz, D. E. Hunka*

## Abstract

Sandia researchers use ion mobility spectrometers for trace chemical detection and analysis in a variety of projects and applications. Technologies developed in recent years based on ion mobility spectrometry (IMS) technology include explosives detection personnel portals, the material area access checkpoint of the future, an explosives detection vehicle portal, hand-held detection systems such as the Hound and Hound II, micro-IMS sensors, ordnance detection, and Fourier transform IMS technology. The emphasis to date has been on explosives detection, but researchers have also pursued the detection of chemical agents.

The IMS-mass spectrometry (MS) combination overcomes several limitations present in simple IMS systems. Ion mobility alone is insufficient to identify an unknown chemical agent. Collision cross section, upon which mobility is based, is not sufficiently unique or predictable a priori to be able to make a confident peak assignment unless the compounds present are already identified. Molecular mass, on the other hand, is much more readily interpreted and related to compounds. For a given compound, the molecular mass can be determined using a pocket calculator (or in one's head), while a reasonable value of the cross section might require hours of computation time. Thus, a mass spectrum provides chemical specificity and identity not accessible in the mobility spectrum alone. In addition, several advanced mass spectrometric methods such as tandem MS have been extensively developed for the purpose of molecular identification. With an appropriate mass spectrometer connected to an ion mobility spectrometer, these advanced identification methods become available, providing greater characterization capability.

## Accomplishments

- *Ion Transport Simulations*

In designing the interface, we conducted several sets of simulations. The goal of these simulations was threefold: (1) to determine the fields necessary to conduct ions from the IMS detector to the intake of the MS; (2) to estimate the fraction of ions that would arrive at the MS; and (3) to optimize electrostatic focusing elements, geometry, and aperture sizes such that the greatest number of ions would be transmitted. We used SIMION to run the simulations.

Our original simulations did not take into account the high pressure (essentially atmospheric) at the front end of the interface. The result of these simulations was that all ions leaving the IMS through a small hole in the Faraday plate could be focused into a fairly small spot size and introduced into the IMS. We discovered that not only did we need to take into account pressure and flow effects, but also space-charge effects. The simulation was valid only at ultrahigh vacuum and low ion current. We based the original interface design on these simulations and accomplished substantial work in constructing the interface before realizing the invalidity of this method.

Next, we wrote external code that would approximate the effect of neutrals on ions using two established methods: collisional cooling and viscous flow damping. In collisional cooling, a randomizer determines when ion-neutral collisions occur as a function of collision cross section and pressure. To get an accurate picture of the ion behavior in the presence of substantial gas pressure, we developed an external program that treats collisions in three dimensions.

- *Interface Design*

We reconstructed the plate with a hole at the center through which ions could pass. These ions are then directed onto the inlet of the mass spectrometer a few centimeters away. We made the interface out of a single piece of Macor, a machinable ceramic. This enabled the potentials to differ on the two sides of the interface.

Although the lens elements helped with ion trajectories through the interface, the easiest solution was to make the MS inlet be the IMS detector, essentially reducing the interface to the physical connection between the systems. The plate that replaced the MS inlet plate has the Faraday plate for the IMS embedded in it. Gas flow through the interface is controlled by three separate flow components. First, the curtain gas leaving the mass spectrometer inlet (which is also the IMS detector) runs at about 6 L/min in the direction of the IMS inlet. Second, the IMS countercurrent (drift) gas runs in the same direction as the MS curtain gas.

Third, the sample carrier gas enters the IMS inlet and moves in the opposite direction as the other two. The IMS drift gas and the MS curtain gas flow in nearly identical paths through the IMS, so it is expected that the curtain gas will not disrupt the IMS function.

- *Timing of MS Based on IMS Peak*

The output of the IMS is time dependent, with peaks eluting over a period of approximately a millisecond. The input of the MS is designed to be continuous; that is, ions are continuously fed into the mass spectrometer. These fundamental timing differences were a significant obstacle during the project. Not only is the signal from the IMS transitory, but if several peaks are present in the IMS spectrum, it is necessary to select one peak at a time for MS analysis. We approached this problem from two directions: software control and hardware control. Hardware control was the best approach for overcoming most difficulties of this interface. We installed a physical gate to allow a gating of ions into the mass spectrometer.

- IMS/MS Integration

The final integration of the two systems shows the transmission of ions created in the IMS to the mass spectrometer to be successful, and we are analyzing spectra.

## Refereed

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# Characterization, Performance, and Optimization of PVDF as a Piezoelectric Film for Advanced Space Mirror Concepts

*52726*

*J. W. Martin, J. M. Elliott, R. G. Tissot Jr., M. C. Celina, R. A. Assink, G. D. Jones, T. Dargaville, P. M. Chaplya, D. M. Mowery*

## Abstract

We successfully completed this project examining materials selection and lifetime prediction of smart polymeric materials for use in novel thin film space based adaptive telescope mirrors. Most importantly, of the range of potential candidate materials, we identified two polymers, a polyvinylidene fluoride (PVDF) homopolymer and P(VDF80-TrFE20) copolymer, as having the greatest potential to perform under space environmental conditions. Based on the laboratory testing we conducted, we concluded that the materials are capable of withstanding significant radiation doses and temperatures of up to 120 °C. However, the materials will be sensitive to atomic oxygen degradation, mostly as surface erosion without major consequences for bulk piezoelectric properties. The materials evaluated are lightweight, thin piezoelectric polymer films based on PVDF that deform in response to controlled charge deposition. For the envisaged applications, any mirror materials that will not be shielded from the space environment will be sensitive to the extreme low Earth orbit (LEO) conditions. Environmental degradation due to large thermal cycling, atomic oxygen, the complete unattenuated solar spectrum, micrometeoroids, and charged particles will be unavoidable. Through simulated LEO exposures, we concluded that useful piezoelectric properties of the two best candidate materials could remain for years. We conducted accelerated aging and characterization experiments, i.e., radiation, temperature, and atomic oxygen exposure effects on piezoelectric responsiveness. Experiments resulted in various critical performance and selection criteria eliminating a range of other potential polymers. For fabrication of actual mirror materials, further optimization and development efforts for processing these materials are necessary, and overall design issues for multilayered or laminated bimorphs need to be addressed. To validate initial designs and material suitability, we finalized experimental designs and made preparations for these materials to be exposed to actual LEO conditions as part of the MISSE-6 orbital flight project. MISSE-6, a collaboration between the National Aeronautics and Space Administration (NASA) and Boeing to assess materials in space environments, is scheduled to fly in 2006.

## Accomplishments

Our previous research established the fundamental issues related to the polymer-piezoelectric properties and limitations of piezoelectric vinylidene-fluoride based polymers in space environmental conditions. This year, we continued experiments exposing the two most promising materials, PVDF and P(VDF80-TrFE20), to simulate LEO conditions. Additionally, we considered various processing issues with the aim of preparing fully optimized piezoelectric polymer films and multilayered bimorphs. We successfully developed a method for preparing large films of P(VDF80-TrFE20) by spin casting. This required optimizing the solvent, solution concentration, and casting conditions. After carefully choosing the annealing conditions, applied voltage, temperature, and time, we achieved poling of these films to render them piezoelectric. Using this method, we prepared films up to 8" in diameter with the same methods theoretically scalable to larger sizes. We also considered strategies to improve surface quality/specular reflectance, and evaluated various methods of coating one surface of a piezoelectric layer with a curable resin to eliminate any imperfections. The feedback from these efforts clearly indicated that processing and innovative fabrication approaches are critical to any final design and manufacturing strategies of large active film components, and further studies to address those issues are essential.

We continued our collaboration with NASA's Glenn Research Center to study atomic oxygen (AO) and ultraviolet (UV) irradiation effects. We exposed various samples to vacuum UV radiation simulating 1, 2, and 3 years in LEO for direct comparison with irradiation exposures conducted at Sandia. We observed surface radiation damage (crosslinking, discoloration) for all films; however, the fundamental piezoelectric properties remaining in the bulk were largely unchanged. We used $^{19}F$ nuclear magnetic resonance

(NMR) spectroscopy to compare samples irradiated with vacuum UV radiation and gamma radiation for the first time to gain a better understanding of the radiolytic changes (radiation chemistry) within the materials. Additionally, we coupled various x-ray diffraction (XRD) experiments with these advanced solid state NMR analyses and differential scanning calorimetry to fully characterize the features and concentrations of different phases affecting crystallinity and morphology in these polymers and, ultimately, to control piezoelectric properties. Such data are critical for lifetime prediction, understanding the fundamental degradation pathways, and may also be useful for improvements in the testing of materials in other areas where highly radiative conditions are of concern, for example, polymers on spacecraft for near sun missions or in terrestrial applications such as polymers for extreme UV lithography.

We investigated the use of polysilsesquioxane (POSS) nanoparticle additives to limit AO erosion in collaboration with researchers at the Air Force Research Laboratory at Edwards Air Force Base. The impetus for these experiments was the recognition of a potentially mission ending issue where the rate of atomic oxygen erosion significantly increases when a polymer is sandwiched between two metallized layers (one of the potential designs for the mirror) due to trapping of the oxygen species. The inclusion of POSS particles should slow the rate of AO induced degradation due to the formation of a protective $SiO_2$ layer.

Preparations for the upcoming MISSE-6 flight experiment project, where materials will be exposed to actual LEO conditions including atomic oxygen and vacuum UV on the International Space Station, are proceeding. We finalized an experimental design and built a first experimental unit including fabrication of a suitable bimorph holder, electronic circuitry for timed bimorph activation, remote optical detection of bimorph position, and long term data acquisition. We tested this prototype at Sandia, and it is currently being assessed as part of the Boeing/NASA preliminary design review.

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# Continuous Flow Detector for Rapid Pathogen Identification

*67049*

*G. J. Fiechtner, B. A. Simmons, E. B. Cummings, A. K. Singh*

## Abstract

The purpose of this project was to explore the development of a low power, portable detector for the rapid identification of pathogens. We chose a microfluidic platform that encompassed an inlet filter/concentrator based on dielectrophoresis, a mixing section designed to selectively tag pathogens with fluorescently labeled antibodies, and an optical detection section.

## Accomplishments

(1) We implemented high resolution particle image velocimetry (PIV) to characterize a new type of microchannel that is constructed via two level etching using our spatially uniform field design approach (patent pending). Here, we obtain images of the velocity through an epifluorescent microscope and obtain the degree of deviation from uniformity. This is critical for the design bandwidth of our filter/concentrators.

(2) We invented a method to design side channels for selectively deflected pathogens.

(3) We successfully separated *Bacillus subtilis* (an excellent model cell for *Bacillus anthracis*) from a mixture of background particles in a three-channel filter section. We characterized the deviation from the intended uniform field design performance and demonstrated the impact on filter effectiveness and leakage. We designed this filter to operate continuously. We also demonstrated a second trap-and-release mode filter design, which trapped particles selectively along an insulating, uniform field designed ridge. After obtaining results using a glass chip, we designed a chip to be constructed from polymer materials.

(4) We developed two mixing concepts, resulting in a patent application. We used rationally patterned regions of vorticity, which can be implemented to transport fluid in a desired pattern and can be employed to achieve rapid mixing at microscales (limited by slow diffusion on most other microchip based detectors).

(5) We demonstrated selective deflection in a channel with a large number of parallel ridges constructed from polymer and glass materials. Although such a design is inherently unstable, we showed that a large operating region is available in which the desired behavior is exhibited. We also studied the influence of channel surface coatings on performance and, in particular, showed how the device performance could be tailored by the coating applied.

(6) We demonstrated selective concentration/filtering along a ridge in a microchannel, ideal for aligning with an optical fiber modified to detect pathogens evanescently using cavity ringdown absorption spectroscopy.

(7) We designed and constructed a prototype chip that incorporates three functions: selective concentration to a filter channel, mixing within the filter channel, and detection downstream. The device is designed to operate in a continuous flow mode.

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# Infrastructural Development for Flexible Network of Devices

*67087*

*T. Q. Thai, S. A. Mulder, A. P. Lydon, M. A. Leger, W. A. Amai, J. Z. Liang, Z. L. Lovelady*

This is a classified report.

# Monolithic Reconfigurable Radio Frequency Microelectromechanical System (RF MEMS) Antennas

*67088*

*L. M. Feldner, C. T. Rodenbeck, C. D. Nordquist, C. W. Dyck, B. H. Strassner II*

## Abstract

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 millimeter-wave antennas, which can be fabricated monolithically 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.

Realizing these advances requires a substantial research and development effort addressing critical RF/microwave antenna and system design issues in conjunction with sophisticated MEMS processing and integration technologies. This project is successfully integrating RF MEMS switches and phase shifters into novel antenna topologies in order to demonstrate proof-of-concept reconfigurable radiating elements, feed structures, and monolithic reconfigurable electronically scanned antenna arrays. Successful fabrication of the first ever completely integrated monolithic reconfigurable electronically scanned antenna array will demonstrate the level of technological maturity appropriate for further development in the future. Specific antenna design goals emphasized miniaturization, radiation efficiency, versatility, and amenability to monolithic and hybrid microsystem integration.

Our work is highly synergistic with other RF MEMS and RF/microwave hybrid miniaturization work at Sandia. Our project continues to leverage ongoing RF MEMS switch and phase shifter development at Sandia, and we routinely collaborate with the Passive Electronically Steerable Array (PESA) LDRD project team. Our miniature antenna research has already directly impacted RF tag projects at Sandia and generated a patent application. In addition, our antenna technologies are of interest to the Systems Analysis of Networked Sensors LDRD project.

## Accomplishments

We fabricated a number of prototype antenna elements and measured them on both conventional RF substrates and custom substrates, including prototypes with monolithically integrated RF MEMS switches. We addressed many process roadblocks to monolithic integration, including the development of new wafer bonding techniques and packaging approaches.

Our electrically small antenna research demonstrated a new class of ultrathin miniature antennas that we are now using in current RF tag applications. Antennas smaller than seven square inches (~1/10 of a wavelength on the longest side) achieved impressive gains (-1 dBi) at 400 MHz in the laboratory. We also demonstrated 400 MHz antennas about the size of a US quarter dollar with -14 dBi gain. We also developed and fabricated high gain, high performance radial endfire antenna arrays for wireless sensor network applications.

We developed a novel wafer-probe monolithic antenna measurement technique in existing anechoic chamber facilities. We used this new capability to measure initial $K_a$ band monolithic reconfigurable triangular patch antenna element prototypes. Experimental and simulated results are in good agreement, and we demonstrated the feasibility of our approach to RF MEMS enabled monolithic reconfigurable antennas.

Accurate full-wave numerical electromagnetic modeling of integrated antenna elements and switch structures led us to the discovery of new operational antenna modes, which we are using in second generation element and array prototypes. We also successfully miniaturized our monolithic antenna elements by an additional 33% while maintaining adequate efficiency (gain drops from 4.7 dBi to 3.1 dBi). We designed single reconfigurable antennas operating in $K_u$ (12–18 GHz) and K (18–27 GHz) bands and plan to fabricate and measure them.

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# Next Generation 3-D Inspection System for Facility Monitoring

*67089*

*J. J. Carlson, T. H. Ko, M. R. Muguira, J. R. Coombs, C. Q. Little*

## Abstract

The goal of this work was to develop next generation two dimensional (2-D) and 3-D inspection technologies for facility and material monitoring. These technologies include components for detection, tracking, and assessment. Our approach involved the integration of pre-existing 2-D and 3-D sensing technologies previously developed at Sandia. These technologies, 3-D real time shape detection (RTSD) and 2-D real time video tracking (RTVT), are used to continuously maintain real time, high fidelity 3-D models of both static and dynamic components of monitored areas. Both systems provide real time estimates of the shape, location, and motion of humans and moving objects. We demonstrated a unique analysis capability currently unavailable in other 3-D sensing technologies to process the 3-D sensor data and to identify activities or conditions that could signify potential threats (e.g., unauthorized intrusions into a restricted space, structural changes in facilities, material movement). These technologies are mature and cost effective, and we have already fielded elements in a number of interior and exterior applications. We can use the 3-D localization capabilities to detect and track special materials in cluttered environments. In addition, we developed and implemented a new and revolutionary wide baseline multicamera self-calibration algorithm. Self-calibration will dramatically extend the practicality and usability of wide baseline multicamera 3-D vision systems, especially in potentially hazardous environments. We also developed an interactive 3-D visualization capability. This visualization allows remote, interactive inspection of a monitored area (via Internet, satellite, or telephone links) using a 3-D computer model of the area that is rendered from actual sensor data.

## Accomplishments

We achieved several standout accomplishments in FY 2005. We integrated, installed, and demonstrated 3-D monitoring and tracking capabilities. We also developed new and revolutionary wide baseline multicamera self-calibration algorithms. These new self-calibration capabilities have the potential to revolutionize 3-D vision sensing applications, especially in hazardous or difficult environments. With these new developments, we can easily and rapidly deploy wide baseline multi-camera 3-D sensing systems.

We advanced two monitoring systems based on passive techniques (3-D RTST and RTVT) to track objects extracted using depth from motion. Both technologies provide cost effective, practical, and reliable sensing, visualization, and 3-D mapping capabilities necessary for real time unattended monitoring. The monitoring systems function in highly variable interior and exterior environments for applications ranging from monitoring mostly static storage vaults to extremely dynamic work areas where movement and human activities are commonplace. These systems can automatically and reliably detect changes in the physical 3-D characteristics of the monitored area, detect and track material movement, and allow a remote operator to visualize, interpret, and assess the detected changes using a 3-D graphical interface.

The RTST system is a full 3-D sensor that provides the 3-D position, volume, and shape of objects detected. It monitors the scene from several points of view and triangulates to locate motion in 3-D. RTST provides the sub-second updates necessary for monitoring humans and moving objects.

A major impediment limiting the practical application of the RTST system was the manpower intensive nature of the calibration process and the inaccessibility of certain areas. Calibration determines model parameters that allow points in world coordinates to be mapped onto camera coordinates (pixels). Current techniques require the corresponding position measurement of calibration points in both world and camera coordinates. The accurate measurement of the 3-D world position of calibration points is extremely tedious and time consuming. In addition, the model is nonlinear and the parameter estimates are sensitive to measurement noise. This fact necessitates the position measurement of large numbers of points. We developed revolutionary, self-calibration algorithms that will dramatically expand the practicality and usability of the 3-D RTST for many existing and new applications. We can demonstrate that the determination of model parameters in a two or more camera system is possible

without any knowledge of the world position of calibration points. The parameters are determined simply by measuring the corresponding pixel positions of calibration points in the field of view of each camera. The process involves moving an easily detectable target through the common field of view of the multicamera system. We can also use opportune targets (e.g., intruders). Using this approach, we can accomplish calibration quickly. Also, we can use future target detections to update or validate the calibration. Since it is easy to acquire a large number of measurements, the parameter estimates tend to be much more stable and accurate. We successfully verified this process using simulation. A major contribution of this work was to validate simulation results using a real system. This effort will revolutionize applications of the 3-D RTST.

# Risk Assessment Meta Tool

*67090*

*A. M. Bouchard, L. J. Whittet, G. C. Osbourn*


## Abstract

The purpose of this project is to import and consolidate data from multiple risk assessment tools that do not "talk to each other" into a single environment, then allow the user to generate new analyses on demand. The first version will allow the user to import multiple files from the joint conflict and tactical simulation (JCATS) and construct on demand analyses from these files. In the second version, we will add the capability to import files from the adversary timeline analysis system (ATLAS) and develop analyses that combine data from JCATS and ATLAS. By the end of the project, we plan to complete a third version to incorporate another file format and additional analyses we have yet to determine. 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 demand. The end result will be a Meta Tool 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.


## Accomplishments

In the first year of this project, we gathered requirements from JCATS specialists, created a conceptual design for the user interface to support the JCATS requirements that can easily be expanded as we incorporate other tools, and designed the underlying self-assembling software (SAS) building blocks to support the Meta Tool.

This year, we completed the detailed design of the user interface, particularly how the user will input an analysis using high level English-like sentences. We also designed the code generation capability that translates the user's inputs into the SAS building blocks that will self-assemble the user's analysis code "under the hood." In addition, we implemented the SAS building blocks and are in the process of implementing the user interface and code generator functionality (exceeding 10,000 lines of code so far). We also implemented a specific analysis that JCATS specialists perform frequently (currently, by hand) as a test case. The code generator does successfully generate the SAS building blocks to complete the JCATS analysis, and those building blocks successfully self-assemble and correctly execute the analysis. We are now extending the code generator capabilities to more complicated analyses and mathematical functions.

The Department of Energy JCATS Users Group expressed great interest in the Meta Tool's JCATS analysis capabilities, in combining JCATS output with ATLAS files, and the results of force-on-force exercises. If the force-on-force exercise results are saved in text files, the first version of the Meta Tool should be capable of supporting combined analysis of JCATS and force-on-force exercises since JCATS files are also text files.

# Featureless Spread-Spectrum Waveform Design and Processing

*67091*

*G. B. Haschke, R. M. Axline, C. R. Collins, R. M. Holman, D. D. Kiffer*

## Abstract

We proposed a new method for designing spread-spectrum waveforms that are devoid of the periodicities found in traditional direct-sequence spread-spectrum (DSSS) modulation and, thus, are significantly less vulnerable to standard eavesdropping detection and exploitation methods.

This year, we expanded the capability of the prototype transmitter and receiver to support advanced error correction techniques, and demonstrated the entire system over a realistic wireless link involving an airborne transponder.

We conducted two vulnerability analyses of both simulated waveforms and those produced by the prototype transmitter: one performed at Sandia and one performed at the New Mexico State University Physical Science Laboratory (PSL). These analyses document the futility of using traditional techniques to exploit these waveforms.

## Accomplishments

In FY 2004, we developed a new methodology for designing DSSS waveforms that are devoid of the periodicities found in traditional DSSS modulation and, therefore, are significantly less vulnerable to standard exploitation methods.

In FY 2005, we extended the prototype receiver algorithms to perform data symbol detection and tracking and data bit error decoding. We demonstrated the complete system prototype by decoding digital information from signals transmitted over a wireless link through a transponder during two demonstrations of the Global Personnel Recovery System (GPRS) project. For the GPRS 2T+1 Mountain demonstration, we located a single transponder on South Baldy Mountain, New Mexico, approximately 80 miles south of Albuquerque. For the GPRS 2T+1 Airborne demonstration, we installed the complete transponder payload aboard a Cessna 175 aircraft flying in an east-west "race track" circuit, approximately 50 miles south of Albuquerque, at an altitude between 8000 and 10,000 feet above ground level. During each of these demonstrations, we transmitted signals in the ultrahigh frequency band, and a transponder relayed the signals to the GPRS tactical ground station in the C frequency band.

We also investigated the compatibility of diamond waveforms with the turbo data encoding-decoding algorithm developed in the Novel Methods for Ultracompact Ultralow Power Communications LDRD project. We used a turbo code to encode error correction for the data portions of the transmissions during the GPRS demonstrations. The chief objective of these experiments was to investigate the ability of turbo error correction encoding algorithms to lower the probability of bit error in messages using diamond spreading codes. We processed the recorded signals to detect preambles and to demodulate the data portion of each message in two different ways: (1) as data symbols (bits) that have no error-correction coding and (2) as data symbols to be decoded by a turbo decoding algorithm. The performance of the turbo encoded waveform with diamond spreading was similar to that of the turbo encoded waveform with binary phase shift keying (BPSK) spreading.

We performed two vulnerability analyses to explore the resistance of diamond waveforms to standard signal exploitation techniques. We based the analysis performed at Sandia on knowing the structure of the signal, which allows the analyst to readily make a quantitative estimate of the signal-to-noise ratio of certain features produced from conventional analysis techniques. At PSL, the analyst only knew that the data records contained samples of three types of waveforms at various signal-to-noise ratios (SNRs). We designed the analysis at PSL to be a "blind" test that indicates the difficulty encountered by a signal analyst attempting to intercept and exploit the waveforms.

In this project, we achieved all objectives, except for developing a conceptual design for the implementation of a transmitter in miniaturized hardware and developing a methodology for coordinating the baseband spreading code algorithm in the transmitter with that in the receiver. These objectives remain important and should be pursued in future efforts.

## Other Communications

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D. Carbajal and T. Hooker, “Waveform Analysis for the Sandia Laboratory,” New Mexico State University Physical Science Laboratory Report PSL-05-28, Las Cruces, NM, 2005.

# Tracking Slow Moving Objects in a GPS-Denied Environment

*67092*

*T. D. Osborn, J. R. Ford, R. Koudelka, M. R. Daily, D. W. Carr, J. T. Feddema, K. A. Neely, J. R. Bryan, J. L. Novak, V. Shulfer, R. E. Abbott, J. R. Podgorski*

## Abstract

Sandia has developed a unique approach to determining the latitude and longitude of an object on the earth based on tidal acceleration vectors. These are signals of opportunity that cannot be shielded or spoofed and are available everywhere on earth. The tidal acceleration vector, which is present everywhere on the globe and causes the ocean tides, arises from the motion of the moon and sun and, to a lesser extent, the planets of our solar system. This acceleration vector varies uniquely as a function of the global position and time. Conversely, the combination of accurate timing and extremely high precision three-dimensional acceleration data yields a specific location on the globe. Sandia has developed numerical models and preliminary hardware to demonstrate this method of geolocation and filed for patent protection of the key concepts.

We undertook an effort to develop an accurate geolocation system employing tidal forces to determine latitude and longitude. Our prototype system is based on the best available commercial-off-the-shelf (COTS) components. Unfortunately, the performance of our system is limited to a localization error of circa 1000 km due to the limitations of presently available COTS accelerometer technology. Our analysis shows that the key enabler for implementation of a useful GeoLocator™ system is the development of a high precision three-dimensional differential accelerometer with exceptional performance in the Hz band. By contrast, presently available COTS portable accelerometers are designed primarily for responsiveness in the higher frequency ranges needed for inertial navigation systems, and performance at ultralow frequencies demands alternative design approaches.

## Accomplishments

The primary objective of this project was to examine the feasibility of developing a geolocation system based on measurement of the solid earth tidal acceleration vector. Such a system would be much more practical to build if commercially available accelerometer technology and electronics could be integrated into a functional system since custom sensor development entails substantial risk and expense. Therefore, one of our chief goals was to explore the performance of the best available COTS portable accelerometers to determine whether unreported capability at ultralow frequencies might be sufficient for some practical geolocation tasks.

Our simulations indicate that the localization capability of a system based on accelerometers with noise performance of -100 dB $G^2$/Hz or greater in the key frequency band near 10 micro-Hz is limited to within thousands of kilometers. Localization in the subkilometer range places even greater demands on accelerometer performance, about -160 to -180 dB $G^2$/Hz at 10 µHz, depending on the statistical characteristics of the model used for 1/f or flicker noise. The extrapolated datasheet performance for the world-class Honeywell QA-series accelerometers is -100 dB $G^2$/Hz at 10 µHz. Our measured data agree with the datasheet except in the downward direction, where surplus noise, attributed to limit-cycle oscillations in the force-balance control loop, yields -75 dB $G^2$/Hz. The performance of applied microelectromechanical systems (MEMS) SiFlex accelerometers in parallel experiments was about -90 dB $G^2$/Hz but was independent of orientation. Thus an accurate geolocation system cannot be built with COTS accelerometers based on noise power spectral density considerations alone.

Exploitation of the tidal acceleration vector remains an intriguing and elegant theoretical solution to the ongoing problem of a global positioning system denied geolocation. Successful implementation of the concept will, however, require the development of accelerometer technology with orders of magnitude improved noise performance in the µHz frequency band. As this frequency regime is not relevant to standard navigation systems and has no other known economic drivers, it is unlikely that commercial industry will provide a solution. Optical accelerometer technology, presently at a very early stage of development, may eventually be capable of adequate µHz performance for geolocation. However, there are fundamental limitations on miniaturization and power consumption of optical accelerometers that would limit the

deployment opportunities of the system. Given these limitations, it is difficult to unreservedly argue for continued development of such a geolocation system. Future work should focus on long period accelerometer development and advances in flicker noise models and signal processing, given that such advances are prerequisite to integration of a practical system.

# Optical Communications

*67093*
*V. M. Hietala*

## Abstract

This project is exploring the feasibility of a unique optical communication method. The project is composed of two primary tasks: proof-of-concept system demonstration of the proposed technique, and a study of the necessary custom components for eventual realization. Last year, emphasis was placed on the system feasibility demonstration. Work this year included a study of component technologies that can be applied to the concept and a more sophisticated system-level demonstration. We believe we have sufficient proof of feasibility to seek follow-on funding from appropriate customers.

## Accomplishments

We assembled a realistic testbed and successfully performed a complete system demonstration using commercial hardware. We completed an end-to-end data transport demonstration and analyzed the results to understand the ultimate performance potential of the concept. We proved the concept feasibility and developed a basic understanding of some of the important design and performance trade-offs that would be required in any future application of the concept. We completed the study of the custom or commercial component options for various versions of the system.

# A Unique Vibration-Based Miniature Power Generator for National Security Applications

*67094*

*R. A. Kellogg, K. R. Pohl, C. L. Davis, J. K. Brotz*

## Abstract

Emerging sensors for national security and health monitoring, as well as communication applications, would benefit from long term reliable electrical power sources. Present battery technologies do not provide reliable power for extremely long life (> 30 years) in sufficiently small sized packages. The need for miniature reliable self-regenerating power systems is necessary, and the development of such devices is practical, utilizing Sandia's microelectromechanical system (MEMS) technology. This project aims to develop a long life, vibration based miniature power generator, or "microgenerator," that scavenges ambient mechanical energy to provide electrical power for integrated circuits and other low power devices through electromagnetic transduction.

The realization of the proposed microgenerator depends on the successful integration of an efficient mechanical vibration pickup system with a planar electromagnetic induction based transducer that converts motion into electrical energy. Development issues with the vibration pickup system include fabrication of a robust low frequency (< 30 Hz), low loss resonator having an embedded array of high strength magnets. Development challenges with the induction based transducer include fabrication of a rugged, multilayer, multiturn planar meander coil having low resistance and optimal inductive coupling. To attain maximum performance of the microgenerator, precision alignment of the vibration and transducer components must be maintained throughout packaging to form an integral unit. Although the engineering challenges and development issues associated with the design, fabrication, and packaging of the microgenerator result in a high risk endeavor, there is a large payoff in realizing a long term reliable power source for national security applications.

## Accomplishments

We completed the design, and the fabrication is in the final stages, to produce a miniature vibration based energy harvester prototype. This was made possible through the development of integrated models and simulations. We established principles of operation of velocity-damped resonant generators, the random vibration environment, electromagnetic transduction, power conditioning, and device optimization. We evaluated the implementation of a novel single magnet transduction design used with a high permeability core and coil to generate electrical power. We explored approaches to increase power output through improved materials, mechanical designs, and electronics.

More specifically, the FY 2005 goal for the vibration based miniature power generator project was to realize a testable prototype. Toward this end, we focused on modeling, simulation, design, and prototype fabrication. We modeled, in detail, the principles of operation of the velocity-damped resonant generator (VDRG). This included an analysis of the single degree-of-freedom oscillator while considering the effects of geometric constraints. We determined the damping power that may be extracted from a resonant mass in terms of key parameters, including mass, density, geometry, vibration frequency, and amplitude. An analysis of random vibration theory and power spectral density information, quantifying different vibration environments, yielded mathematical relationships describing harvestable power. We evaluated different transduction methods to convert mechanical to electrical power. We rejected a planar meander coil approach, originally the basis for the project, due to fabrication issues and low projected power outputs.

We developed a high permeability core electromagnetic transduction concept as a means of increasing the number of coil windings over a larger volume containing moderate magnetic flux changes. This approach allowed sufficiently large voltages to be produced for microelectronics applications. An enabling concept, utilizing a single magnet for the high-permeability core approach, capitalizes on magnetostatic forces and enables device operation in low vibration environments. We developed electric power conditioning circuitry to provide voltage multiplication, rectification, and energy storage for microelectronic applications. We constructed P-Spice models to guide design optimization of the power conditioning electronics for a vari-

ety of input conditions and loads. To facilitate the design of a prototype energy harvester, we analyzed all systems including the transduction elements, mechanical components, and electronics. We found that magnetic hysteresis, eddy current, air damping, and resistive and diode drops all contribute to significant but manageable power losses. Ultimately, the modeling and simulation efforts facilitated the first-order optimization and design of a VDRG prototype and shed light on methods to increase voltage and power outputs from smaller volumes. Based on this modeling work, we designed a single magnet prototype. It employs simple components with generous tolerances to facilitate manufacture using a combination of conventional machining and lithographically defined metal (LIGA, for the German term *Lithographie, Galvanformung, und Abformung*, for lithography electroforming, and molding) processing techniques. If our projections hold, we can harvest 200 microwatts or more of electrical power (from a volume of 12.6 $cm^3$) from a 60 Hz vibration of well under 1 g.

# Vulnerability Assessment with Dynamic Reverse Engineering of Embedded Processors Through Innate Debug Mechanisms of System-on-Chip Integrated Circuits

*67096*

*J. J. Clement, A. Phan, S. Montague, J. M. Green, J. L. Etzkin, E. I. Cole Jr.*

## Abstract

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 (ROM), and random access memory (RAM). 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 developed to test interconnections between ICs on a printed circuit board. The Joint Test Action Group (JTAG) has specified an Institute of Electrical and Electronics Engineers (IEEE) standard for boundary-scan testing. By adding to the five commands required by the standard, the IC designer can enhance the 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.

Sandia desires to be able to perform focused reverse engineering of SOCs by leveraging our current analysis capabilities and developing new innovative techniques to quickly identify the JTAG interface pins. Further tracing to the JTAG controller may be needed if the JTAG is disabled for security. Re-enabling the JTAG will require solutions to bypass both physical and electrical security mechanisms.

With the pins identified and the JTAG enabled, we can 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 the ability to focus on critical areas. The productivity enhancement with this capability will be immense. In-system programming will allow us to read ROM and RAM memory contents, a capability that currently is time consuming and destructive.

## Accomplishments

We developed and demonstrated a tool based on a commercial system that is capable of communicating over the JTAG interface. We also made promising progress on developing a methodology to identify JTAG pins on unknown SOC ICs. The IEEE standard requires only four or five pins to implement JTAG, and SOC ICs may have many hundreds of pins. The details of these accomplishments are classified.

# Nonlinear Optical Detection of Biological and Chemical Aerosol Agents Using Femtosecond Lasers

*67098*

*T. R. Nelson, J. Urayama, M. L. Naudeau, E. A. Disch, T. S. Luk*

## Abstract

Nonlinear 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 standoff 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. It will also 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.

We can use propagation control strategies employing short pulse laser pulses to controllably generate intensity at range to initiate nonlinear spectroscopic processes. By exploiting optical nonlinear effects such as multiphoton excited fluorescence, third harmonic generation in air, stimulated Raman scattering, and laser induced plasma breakdown in aerosol droplets, it is possible to enhance backscatter signatures for improved collection. It is also possible to 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. We will conduct proof-of-principle laboratory experiments to evaluate the feasibility of nonlinear remote sensing. We will use calibrated experiments of surrogate systems to baseline performance models and assess operational capability.

## Accomplishments

We met all FY 2005 milestones. We constructed range dependent performance models for multiphoton excited fluorescence (MPEF) based on previous experiments in basic biological constituents. We examined riboflavin, tryptophan, and nicotinamide adenine dinucleotide (NADH) using 800 nm radiation. In addition, we measured fluorescence signatures in *Bacillus globigii* and *Bacillus megaterium*, which are surrogates for anthrax. We performed similar measurements for thiodiglycol (TDG) and pinacolyl methylphosphonate (PMPA), which are surrogates for mustard gas and soman, respectively. We performed experiments to examine the use of third harmonic generation (THG) in air for the linear excitation of fluorescence, as an alternative to MPEF. Calibrated measurements demonstrated saturated conversion efficiencies of 0.1% using an 800 nm source, and > 0.1% using a 1550 nm source, without saturation. Furthermore, the data illustrate a strong dependence on the focusing condition, favoring higher f-numbers, which suggests an advantage in range applications. We performed experiments to demonstrate the simultaneous excitation of multiple biological materials using THG in air from a tunable femtosecond laser source, demonstrating the ability to control the material excitation with the possibility of being able to measure relative concentrations.

We designed and are still developing preliminary experiments to measure calibrated spontaneous and stimulated fluorescence cross sections from biological surrogates. We began examining specifics of target morphology (droplet, spore, aggregate, plume) and its impact on signature phase function and yield. We designed and constructed a compact white light fiber source to begin experiments exploring broadband differential adsorption laser applications in the near and mid-wave infrared. We are currently characterizing the spectral, temporal, and spatial phase properties of this source in the laboratory. We are also designing experi-

ments to measure the impact of atmospheric propagation on these parameters. We constructed an experimental setup to perform stimulated Raman scattering (SRS) and coherent anti-stokes Raman spectroscopy (CARS) measurements on biological and chemical surrogates in the fingerprint region (1000–3000 $cm^{-1}$). We obtained SRS signatures in benzene (standard reference) and TDG. We obtained CARS signatures in benzene, and experiments are under way on dipicolinic acid (DPA), which is present in anthrax spores. We constructed second generation adaptive pulse shapers with ~10 $cm^{-1}$ resolution for use in CARS experiments. We constructed performance models for CARS, which suggest the potential of a clear advantage in performing nonlinear spectroscopy with an infrared pump laser. We performed further pulse shaping experiments on materials with known MPEF resonances that demonstrated a possible phase dependence, which may not be strictly dependent on the peak laser intensity.

# Polymer Electronic Devices and Materials

*67099*

*D. A. Chinn, B. L. Silva, T. M. Wilson, K. Rahimian, F. P. Doty*

## Abstract

Polymer electronic device performance is limited by high resistivity in conductors and low resistivity in semiconductors. By increasing local order, thus reducing trap density, performance is improved in both materials.

We developed a new conducting ink blend using polythiophene as a hole conductor, carbon nanoparticles as an electron conductor, and an organic acid as a dopant. The material has a resistivity of 0.014 ohm-cm, 800x better than any other polymer we measured. It was also among the best demonstrated in an air-stable organic ink, enabling an ink-jet printed diode with a current over 1 microamp at 5 V. To further improve conduction, we performed acid and hydrogen treatments on carbon nanotubes, which altered their behavior versus untreated nanotubes. Raman results do not show any difference from control nanotubes, indicating an endohedral reaction.

Hall measurements of dimethyloctyloxypolyparaphenylenevinylene (D1PPV) with varying nickel contents indicate that metal content below 11 ppm is essential for achieving the high resistivities required of semiconductors. To improve mobility, molecular alignment by stretching cast films yielded 3:1 stretch ratios, but x-ray diffraction studies thus far were uninformative due to thin films. The photoconductive response of D1PPV shows that charge collection is improved when illuminated with subgap light. We demonstrated the ability to build active devices on a variety of substrates using very simple methods such as painting, and built a flexible diode using only strips of cloth soaked in various conducting polymers.

## Accomplishments

Work focused on material improvements by reducing resistivity for conducting polymers used in diodes and increasing resistivity in semiconducting polymers for increased charge collection efficiency in neutron detectors by reducing trap density.

We developed a new, highly conducting, all organic material based on the concept of a bulk heterojunction by combining a dopable conducting polymer, carbon nanoparticles, and an organic acid in common solvent blends. We applied this “synthetic metal” to flexible diodes made entirely by ink jetting. The diodes consist of a stack of polyaniline sulfonic acid, D1PPV, and finally the synthetic metal. This device yielded a forward bias current well above 1 microamp at 5 V, replacing an earlier diode stack that had one order lower current at best.

To build these devices, we completely rebuilt an ink jet printer and added a heated jet, allowing us to write lines (~500 microns) and spaces (~200 microns) of polymeric inks over slightly contoured surfaces. These devices are flexible and are on plastic, glass, and printed circuit board materials. We also developed ink jetting techniques for shellac, used as a dielectric that does not dissolve underlying layers. These inks can be applied using paint brushes, creating devices on almost any surface. We also demonstrated a diode made of cloth and coated nylon fibers with these polymers, showing that plastic electronic devices can be ubiquitous in our surroundings and do not necessarily have to look like electronic devices.

To further enhance the conductivity of synthetic metal, we treated single walled carbon nanotubes in hot hydrogen, sulfuric acid, and perchloric acids to improve the solubility of the nanoparticles in organic solutions. Acid treatments remove metal catalysts and end groups. The treatments altered the behavior of the nanotubes, but Raman spectroscopy showed no change, indicating that the acid remains inside the nanotubes. We expect nanotube based inks to have lower resistivities than C60 based inks, as low as 100 ohm/sq. Our standard mixture of synthetic metal with equal weights of all three solid ingredients yields a resistivity of 0.014 ohm-cm, or a conductivity of 71 S/cm, compared to other conducting polymers such as PEDOT, a polypyrrole, and sulfonated polyaniline that measure between 11 and 45 ohm-cm.

Measurements on a 5 micron gap van der Pauw structure indicate that metal content in semiconducting polymers is a critical factor to control. At 11 ppm nickel, D1PPV yields a resistivity of $2.1 \times 10^7$ ohm/sq, where increasing the metal content to 53 ppm and above yields resistivities of $9.2 \times 10^6$ ohm/sq and lower. High resistivities are essential to limit leakage

currents, which improve charge collection efficiency in polymeric neutron detectors by allowing for higher fields to be applied across a film. Hall measurements on synthetic metal give conflicting results on carrier type (hole or electron), indicating that it may be an ambipolar conductor.

We measured the pulsed photoconductive response of D1PPV with and without a sub gap infrared illumination. These materials have very high trap densities ($> 10^{17}/cm^3$), and this result shows that carriers can be excited out of trap states into the conduction band with sub gap light, increasing charge collection efficiency in devices such as neutron detectors.

Mobility in organic semiconductors is several orders lower than inorganic semiconductors, so we are stretch aligning various poly(paraphenylene vinylene) polymers by casting films on Teflon tape, then stretching the tape when the film is dry. The use of plasticizers aids in stretching, but will likely interfere with electronic performance. Light scattering measurements show that high molecular weight (624,000) D1PPV has a radius of gyration at 195 nm, indicating that the molecule is somewhat open in dilute solutions, allowing for stretching with the right solvents.

# Small Circuits for Cryptography

*67101*

*T. J. Draelos, R. C. Schroeppel, M. D. Torgerson, R. D. Miller, C. L. Beaver, W. E. Anderson*

## Abstract

This project examines a number of hardware circuit design issues associated with implementing certain functions in field programmable gate array (FPGA) and application specific integrated circuit (ASIC) technologies. We researched circuit designs for the advanced encryption standard (AES) and secure hash algorithm (SHA)-1 that have an extremely small hardware footprint, yet show reasonably good performance characteristics when compared to the state-of-the-art designs found in the literature. Our AES performance numbers are fueled by an optimized composite field S-box design tuned to the Stratix chipset. Our SHA-1 designs use register packing and feedback functionalities of the Stratix logic element FPGA (LE FPGA), which reduces the logic element usage by as much as 72% compared to other SHA-1 designs.

## Accomplishments

We developed new design strategies applicable to the AES encryption algorithm and the SHA-1 hash algorithm. The resulting circuits have an extremely small hardware footprint, yet do not overly sacrifice speed. The developed optimized S-box composite field technologies led to the smallest Stratix FPGA implementation for AES encryption with an integrated key scheduler. The same designs applied to ASIC technology reduce an AES S-box from 909 gates, using table lookups, to 371 gates in CMOS7.

Our SHA-1 architecture is optimized for the Stratix chipset. We employed register packing and feedback functionalities for optimal performance and size. This reduces logic element usage by as much as 72% compared to other highly optimized designs found in the literature. Our architecture provides the best throughput/design cost ratio over any design found in the literature.

In our analysis, we conducted a comparison of several SHA-1 cores. We restricted ourselves to the Stratix, APEX, and ACEX device families since they all shared a similar logic element structure. The APEX and ACEX logic elements do not incorporate register feedback and use an older manufacturing process; therefore, an implementation on these devices will be slightly larger and slower. We compiled our design using Altera's Quartus II 5.0 software with a speed grade set to 5. To ensure design accuracy, we used the Quartus chip editor as a comparison.

Among the compared architectures, our design had the smallest resource utilization. We reduced logic element resources by as much as 72%. Our data throughput was slower than the other designs since our Round Mix operation takes five clock cycles rather than one.

In addition to the above comparisons, we also compared the throughput/design cost ratio for each of the above designs. Assigning cost metrics C_LE and C_M4K to logic elements and 4 kilobyte random access memory units (M4K), we can compute the resource cost of a design (DesignCost) using the formula:

DesignCost = (# LEs) * C_LE + (# M4K) * C_M4K.

Out of all the designs, our SHA-1 architecture showed the best throughput/design cost ratio whenever C_M4K < 35 * C_LE. Considering that a Stratix EP1S40 device contains 183 M4K RAM blocks, this condition should hold true for many designs.

# Transceiver for Ultralow Power Wake-up and Command

*67104*

*J. A. Payne, C. T. Rodenbeck, R. C. Ghormley II, R. Chanchani, M. D. Sena, K. T. Stalker, M. B. Murphy, K. W. Plummer, S. J. Leslie*

## Abstract

We designed, built, and tested a transceiver for ultralow power wake up and command. The resulting work makes an energy scavenging, ultrasmall data collecting radio frequency (RF) node available to applications where small size and longevity are paramount. FY 2004 activities included the evaluation of several receiver topologies, including, among others, low-intermediate frequency with fast oscillator turn on and surface acoustic wave correlator techniques. In addition to the evaluation of ultralow power RF hardware, we explored system concepts that allow exceptional life extension in an asymmetric communications link. We also prototyped concepts to enable the antenna size to be dramatically reduced. We explored energy harvesting techniques with a particular emphasis on solar recharge through a hybrid photovoltaic/battery system with extremely low background energy needs. FY 2005 activities primarily involved the integration of the techniques developed in FY 2004 into an ultraminiature transceiver. We explored ultrathin FR4 laminate and used it as a low cost, flexible substrate to integrate the devices. We evaluated a number of photovoltaic cells in low light conditions with two classes emerging for integration into the transceiver. We successfully produced and tested a first version integrated board. Additionally, we extensively evaluated the solar power scavenging subsystem before we made a final cell choice for a second integrated transceiver.

## Accomplishments

- Accomplishment #1: Transceiver miniaturized and integrated as flexible data gathering node

Ultrasmall size was one of the primary design goals of the transceiver. Kapton based flex circuits are a traditional choice for thin substrates and are attractive not only for their minimum thickness but also for their flexibility. As we use thinner and thinner Kapton circuits, fragility becomes a key issue. Instead of using the more complex and expensive Kapton based flexible circuit structures, we explored thin FR4 laminates. Modern printed circuit board (PCB) manufacturers routinely use FR4 substrates as thin as 2 mils in multi-layer PCBs. Used separately, these layers are much less than 10 mils thick and become physically flexible with bend radii approaching 1 inch. Very fine lithography processes are still available but at a much reduced price compared to Kapton processes. In addition, the thin FR4 boards exhibit an optimal flexibility for component population, handling, and deployment. We constructed double sided boards from this material and successfully used them for construction of the transceiver. We also constructed four-layer FR4 boards to explore lamination issues associated with thin FR4. In cooperation with the PCB manufacturer, we constructed an 18 mil four-layer thick FR4 board and successfully fielded it, yielding a technique that produces thinner and more mechanically stable boards than the equivalent Kapton laminates. We used four reconfigurable analog to digital converter channels to monitor the health of the photovoltaic/battery system and transmit these data to a nearby receiver.

- Accomplishment #2: Ultralow sleep implemented

Creating a highly asymmetric communications channel enables the integrated transceiver to maintain a standby current of only 10 µA while providing availability to a querying device in less than four seconds. All circuitry is powered down during this sleep mode, including all interface circuitry. A clock wakes the device every 4 s, after which a 300 ms startup oscillator is activated and a receiver samples the in-band RF energy. If no energy is present, the device powers down within 1 µs of waking and waits an additional 4 s before scanning again for RF activity. In the presence of RF, the device fully awakens and performs its programmed function.

- Accomplishment #3: High performance photovoltaic cells optimized for use in low light

Lighting conditions are of particular concern for solar scavenged powered devices, and maximizing use of the solar resource is critical. We designed the power requirements so that twilight conditions would allow the transceiver to continue to function normally. We explored physically flexible photovoltaics to support a flexible circuit board. We evaluated the performance of

thin film, roll printed, flexible photovoltaic cells under extremely low light and found them to be inadequate to power the transceiver. We found mechanically extracted ultrathin photovoltaic strips to perform sufficiently at low light and exhibit sufficient mechanical flexibility. However, they fail under twilight conditions. We found thin silicon photovoltaic cells produced by Amonix, Inc., to perform exceptionally well in twilight conditions and also to be sufficiently flexible. An additional approach to use small size, rigid, triple-junction photovoltaic cells in a "piecewise flexible" fashion also proved satisfactory.

- Accomplishment #4: Ultralow power management circuitry implemented

The system current consumption in most conventional hybrid photovoltaic/battery powered devices usually dominates the power budget. In this solar powered transceiver, the background current consumed sets the device lifetime, so this challenging minimization is critical. We maintained optimal device voltage for less than 3 μA in background current using several techniques. We used ultralow voltage drop Schottky diodes, where the trade-off between reverse leakage current (from the battery to dark photovoltaics) and forward voltage drop (from the photovoltaics to the charging circuitry) is balanced. We used best in class 1.2 μA, 160 mV low dropout voltage regulators to condition the power arriving from the photovoltaic array to the battery and from the battery to the transceiver.

# Reconstruction Algorithm Development and Assessment for a Computed Tomography Based Spectral Imager

*67107*

*B. K. Ford, J. S. Salazar, M. P. Wilson*


## Abstract

The collection of spatial and temporal information is of primary importance for wide field transient event detection. Full field imaging systems coupled with high frame-rate detectors readily enable such collections; however, these configurations are not easily adapted to secondary missions with more stressing spectral requirements. While we can introduce spectral filters (e.g., filter wheel), the spectral bandpasses must be fairly broad to accommodate shorter integration times. For staring mode sensors, chromotomographic systems offer significant advantages over more conventional systems (filter wheels, acousto-optic tunable filters, liquid crystal tunable filters). Most notably, due to the integration of a signal at each pixel, hyperspectral data can be collected with higher temporal and spectral resolutions at higher signal to noise ratios. These instruments are applicable to diverse fields such as optical testing, biomedical imaging, and airborne remote sensing. The goal of this research was to study the applicability of chromotomographic spectrometry (CTS) systems to scene and target characterization from space based platforms.


## Accomplishments

We produced a set of tools that we can use to assess the applicability of nonconventional spectral imaging systems to missions associated with space based optical sensors. During FY 2005, we set out to (1) complete development of advanced CTS reconstruction algorithms, (2) finalize quantitative metrics for anomaly detection and bulk classification, (3) compare algorithm performance using these metrics, and (4) assess the sensitivity of the algorithms to a set of defined error sources. We completed all major milestones except for the fourth goal, which we only partially met.

We completed scene simulation work that involved the modeling of static source signatures for anomaly detection experiments and the production of classification maps associated with previously generated simulated scenes for bulk classification testing. In addition, we developed and evaluated new reconstruction algorithms incorporating filtered image constraints. We found the filtered constrained algorithms to outperform their nonfilter constrained counterparts in terms of both spatial correlation and mean spectral error. While these are important parameters for assessing strict reconstruction accuracy, they do not hold the answers to questions most likely posed by end-users. Specifically, they do not include any information regarding how the data will be used, or better yet, whether or not the data are accurate enough to meet specific mission requirements. To this end, we looked at two possible mission scenarios.

The first scenario assumes a need to detect static point targets against a cluttered background. Our interest was in quantifying the ability of various reconstruction algorithms to accurately estimate anomalous spectra within a scene. We also assessed the dependence of these reconstructions on (1) the number of personal computers used in reconstruction constraints, (2) the ratio of target to background signals, and (3) the correlation between the target and background signatures. For all point target experiments, the projection onto convex sets (POCS) and POCS filter constrained (POCSFC) reconstruction algorithms produce the minimum spectral angles. The subspace constraint algorithm (SCA), on the other hand, performs poorly for the targets but very well for the background spectra.

The second scenario involves the need to perform bulk classifications on all materials within the scene. This would correspond to fairly general classes such as vegetation, water, and bare soil. Specifically, our interest was in quantifying the difference between classifications maps derived from a standard hyperspectral instrument (no reconstruction required) versus those derived from the CTS system. To date, the concept of quantifying a match between classification maps has received comparatively little or no study. We thus proposed and executed a novel approach for quantifying the “optimum” match between classification maps. We performed this analysis in two parts, including reconstruction algorithm performance assessments without, and then with, additional perturbations due to system noise or sensor jitter. We demonstrated that bulk classification performance steadily improves for the POCS

based approaches starting from the POCS to the POC-SFC and a more integrated approach to filter constraining POCS (POCSPCFC) algorithms. The same is true for the SCA based approaches, SCA and SCA filter constraints (SCAFC). These results are consistent with the design and implementation assumptions for these algorithms. However, it was not necessarily clear if one stands out as the better of these two reconstruction approach subsets POCSPCFC and SCAFC. Using both distribution-free clustering (DFC) and k-means based quality metrics, we found that the performance results achieved from the jitter analysis reflect a very regular improvement corresponding to decreasing jitter. These results correspond to our expectations of decreasing performance with increasing sensor jitter. We note that the results tend to be more tightly packed for the DFC based approach. In addition, the results achieved from this analysis generally indicate that as the signal-to-noise ratio increases, performance increases with similar relative changes between algorithms.

# Dispersive Diffractive Optical Elements for the Infrared

*67109*

*S. A. Kemme, R. Boye, A. A. Cruz-Cabrera, E. A. Shields, J. R. Wendt, D. W. Peters, S. Samora, T. Carter*

## Abstract

We will develop two new diffractive optical element (DOE) technologies: (1) a broad wavelength band effective antireflective (AR) structure and (2) a design tool to encode polarization and dispersion information into a unique diffraction pattern. Development of these technologies will enable next generation detection and spectral characterization of fast, transient optical signals and will provide the components required to develop protection systems for the nation's most valuable space assets.

The first technology is an AR layer that may be etched into the DOE surface. For many wavelengths of interest, transmissive silicon DOEs are ideal. However, a significant portion of light (30% from each surface) is lost due to Fresnel reflection. To address this issue, we will implement a subwavelength surface relief structure that acts as an effective AR coating and is tolerant to varying wavelengths and angles of incidence.

The second DOE component technology is a design tool to determine the optimal DOE surface relief structure that encodes the light's degree of dispersion, and possibly polarization, into a unique spatial pattern. Many signals of interest have unique spatial, temporal, spectral, and polarization signatures. The ability to disperse the signal into a unique diffraction pattern would result in improved signal detection sensitivity with a simultaneous reduction in false alarms.

We will also investigate the modeling, design, and fabrication of subwavelength birefringent devices for polarimetric spectral sensing and imaging applications. Polarimetric spectral sensing measures the spectrum of the light and polarization state of light at each wavelength simultaneously. The capability to obtain both polarization and spectral information can help develop the target/object signature and identify the target/object for several applications in nonproliferation, military capabilities, and national security.

## Accomplishments

In Task 1, we numerically investigated the effect of a binary grating etched into silicon. Rigorous coupled wave analysis models the orders reflected and transmitted from the grating. It also models the effect of the grating over a wide range of grating periods, from subwavelength to much larger than the wavelength. With this numerical design tool, we identified a broad wavelength band and an angular band triangular profile that performs well as an effective AR coating. This triangular profile can be fabricated with a wet etch process, which is simpler than dry etch processes, to add to an existing diffractive optic with nonplanar surfaces. We fabricated components using this process and measured their transmission and reflection properties. Based upon these results, we modified the subwavelength period for the wet etch process and completed a second design-fabricate-measurement cycle with improved results.

Also in Task 1, we encoded an iterative Fourier transform algorithm (IFTA) for diffractive optical elements. The application considered here is the conversion of a uniform beam of light to a desired intensity distribution in the far field. Elements that perform this operation are often called computer generated holograms (CGHs). The goal of CGH design is to calculate the height profile such that, in the far-field, the modified beam of light has an intensity distribution matching the desired pattern. We developed and encoded several algorithms that produce an arbitrary pattern, with good uniformity, and the desired dispersion as a function of wavelength. Thus we achieved the goal of a CGH design that would result in a desired pattern that is wavelength dispersed. Our secondary goal of encoding a pattern that would discriminate one polarization from another is attained through a two-step process. First, the CGH designs that produce the intensity patterns for each polarization are realized. Then, the pixels are made sensitive to one of the two polarizations with an additive, subwavelength etch.

In Task 2, we first defined the optical birefringence and dispersion of the subwavelength photonic device with the goal that the two polarizations (transverse electric and transverse magnetic) can be completely separated into two angular ranges. We developed a numerical design code that calculates the effective index versus wavelength for two polarizations (dispersion and birefringence). We developed the fabrication conditions for nanoscale photonic structure in GaAs

with newly installed chemically assisted reactive ion-beam etching equipment. We achieved well controlled vertical features with aspect ratios of 10:1. We designed and fabricated a subwavelength device with angular dispersion for the wavelength range of interest and for the two polarizations.

## Refereed

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# Atmospheric Propagation of THz Radiation

*67111*
*R. J. Foltynowicz*

## Abstract

Terahertz (THz) technologies are proposed as solutions to many current problems, such as remote sensing of bio-chem agents and explosives, covert terrestrial and space based communications, and cold-body tracking. While the promise for THz looms large, we need to resolve many issues for full exploitation of this frequency range. Some address the technical immaturity of the technology in this frequency range, including the lack of compact tunable sources, detectors, and other standard optical components. Besides the hardware problems, there are three very fundamental issues: (1) What is the atmospheric transparency in the THz frequency range? (2) How do we separate desired signals from the thermal blackbody-background created by any warm object present in the THz? and (3) What are the signatures of materials between 0.1 and 10 THz? In this project, we will address the first question regarding the atmospheric transmission to determine in what instances the other two should be examined. In the field of atmospheric THz propagation, there exists a wealth of simulated transmission spectra. Experimental data show that the peak positions of molecular absorption are accurate, while the absorption coefficients at the peak positions are thought to be fairly accurate. However, the background absorption coefficients between the absorption lines are empirical and of unknown accuracy. For many of our applications, such as cold-body tracking, the transmission windows are very important. Thus, verifying this information is of critical importance.

## Accomplishments

We made significant progress in FY 2005 and completed all of our intended milestones for this project. We completed building an operational gas sample cell that we can use not only to conduct THz measurements of atmospheric gas samples, but also any solid, liquid, and gas materials of interest. The cell is rated to conduct experiments for both ambient and high (200 °C) temperatures. In addition, we improved the performance of our THz spectrometer. We converted our detection system from two independent photodiodes to balanced photodiode detection. As a result, our net signals in the time domain went from microvolts to millivolts. Also, we determined the detection limit of our spectrometer, utilizing methanol vapor as the sample. The detection limit of our THz spectrometer is 3 ppm. We completed the THz absorption measurements of atmospheric samples at relative humidities of 18% and 27% in a spectral range of 0.6 to 2.6 THz. We conducted the experiment in a laboratory setting. The path length of the measurement was 1 meter at a temperature of 23 °C and an atmospheric pressure of 630 Torr. The results show that generally, as the relative humidity is increased, the water absorption of THz radiation increases.

# High Confidence Estimation of Relative Event Locations from Space Based Sensors

*67113*

*K. M. Simonson, S. Drescher, F. R. Tanner, E. N. Reckase, M. M. Moya, E. A. Shields, B. R. Funkhouser*

## Abstract

The ability to measure the relative positions of different events is a critical part of the intelligence, surveillance, and reconnaissance (ISR) mission. For long range sensors with limited capability for absolute geolocation, accurate relative positioning is generally achieved only for events that occur simultaneously. The completion of this project enables accurate and precise estimation of the relative locations of events that are not temporally coincident.

We built the technical approach on recent advances in statistics based methods for image cross-registration. A Sandia technique developed in this project is unique in providing a well defined measure of the statistical uncertainty associated with the solution; poor solutions are automatically recognized, minimizing the opportunity for errors to propagate. The approach demonstrates subpixel levels of precision in challenging applications.

In addition, we conducted a rigorous statistical characterization of a specific sensor and all of its data collection modes. We documented the resulting sensor model and implemented it in a software testbed. We developed an end-to-end software demonstration system, which combines the sensor modeling and image registration work. We used this system to show substantial improvements over current performance using real data.

## Accomplishments

Our research advanced along two tracks, image registration and sensor characterization. We completed a demonstration system that combines contributions from each track into an end-to-end capability that shows strong promise for future operational utility.

Accomplishments in image registration:

(1) We matured the technique for edge based image registration to subpixel levels of precision. We documented the algorithm and filed a technical advance on the technique.

(2) We defined and implemented a new technique for edge detection that is well suited for use with the image registration technology. The “local edge detector” is based on the familiar Canny edge detector, but uses a locally defined measure of edge strength, which allows for the detection of structural edges in scenes that include substantial contamination from cloud cover or other phenomena.

(3) We completed a software package that incorporates the local edge detection and image registration techniques.

Accomplishments in sensor modeling:

(1) We developed a sensor model that incorporates contributions from all known signal and noise sources. We based the model on a physical understanding of the sensor and developed it using a substantial training data set.

(2) Based on the sensor model, we developed a strategy for estimating the magnitude of contributions from each source in new images as a function of quantities that are readily measured. This allows for automatic correction of incoming frames; components (artifacts) that are not due to scene structure can be estimated and removed.

(3) We completed a software package for correction of arbitrary sensor frames. The output frames are minimally influenced by sensor and noise artifacts, and are suitable for input to the edge detection and image registration algorithms.

Accomplishments in developing the demonstration system:

(1) We completed an end-to-end software demonstration system, which combines the codes described above. With this system, raw frames are corrected and then passed to the registration codes for solution.

(2) We completed a system performance assessment that shows that this technology may have substantial utility to various existing and potential future programs.

## Other Communications

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# Critical Infrastructure System of Systems Assessment Methodology

*67114*

*J. M. Phelan, P. E. Sholander, B. J. Smith, J. E. Stamp, G. D. Wyss, J. J. Danneels, P. L. Campbell, G. B. Varnado, R. C. Parks*

## Abstract

Assessing the risk of malevolent attacks against large scale critical infrastructures requires modifications to existing methodologies because existing risk assessment methodologies consider physical security and cyber security separately. Current approaches do not accurately model attacks that involve defeating both physical protection and cyber protection elements (e.g., hackers turning off alarm systems prior to forced entry). The risk assessment methodology developed with this effort accounts for both physical and cyber security while also preserving the traditional security paradigm of detect, delay, and respond and accounting for recovery or mitigation of a successful attack before serious consequences occur.

The methodology provides a means for ranking those assets most at risk from malevolent attacks. The automated methodology allows the analyst to play "what if" with mitigation measures to gain a better understanding of how to best expend resources towards securing the facilities. It is simple enough to be applied to large infrastructure facilities without developing highly complicated models. Finally, it is applicable to facilities with extensive security as well as those that are not as well protected.

We developed a general construct to combine information about the consequences of concern for the facility under assessment, the asset failures that could lead to those consequences, the capabilities of the adversary attacking the facility, and the protective features present at the facility to produce risk estimates. The vulnerability assessment portion of the model makes use of existing physical protection system (PPS) effectiveness assessment tools by adjusting the performance of protection elements that are cyber controlled to account for the likelihood that an attacker could penetrate the cyber protection elements of the system. We developed a cyber protection effectiveness assessment approach to support vulnerability estimates for attacks that include cyber elements.

## Accomplishments

We developed hypothetical case studies to use for initial validation and refinements. We modified the case studies developed in FY 2004 and used them to validate elements of the methodology. We re-assessed facilities that Sandia previously analyzed with RAM-W using the integrated methodology and looked for differences and similarities. We identified an energy and water facility to use as case studies. We conducted integrated risk assessments on the identified facilities and refined the methodology based on lessons learned from conducting the assessments.

We began automating portions of the methodology that are appropriate and complete. We developed a high level use-case diagram, system criteria, and a software engineering process to guide the development of the physical/cyber security analyst software. We created and linked alpha versions of the consequence definer, protective measures definer, threat definer, and vulnerability analyst modules. We documented the completed risk assessment methodology.

# Inflatable Antenna with Adaptive Actuators

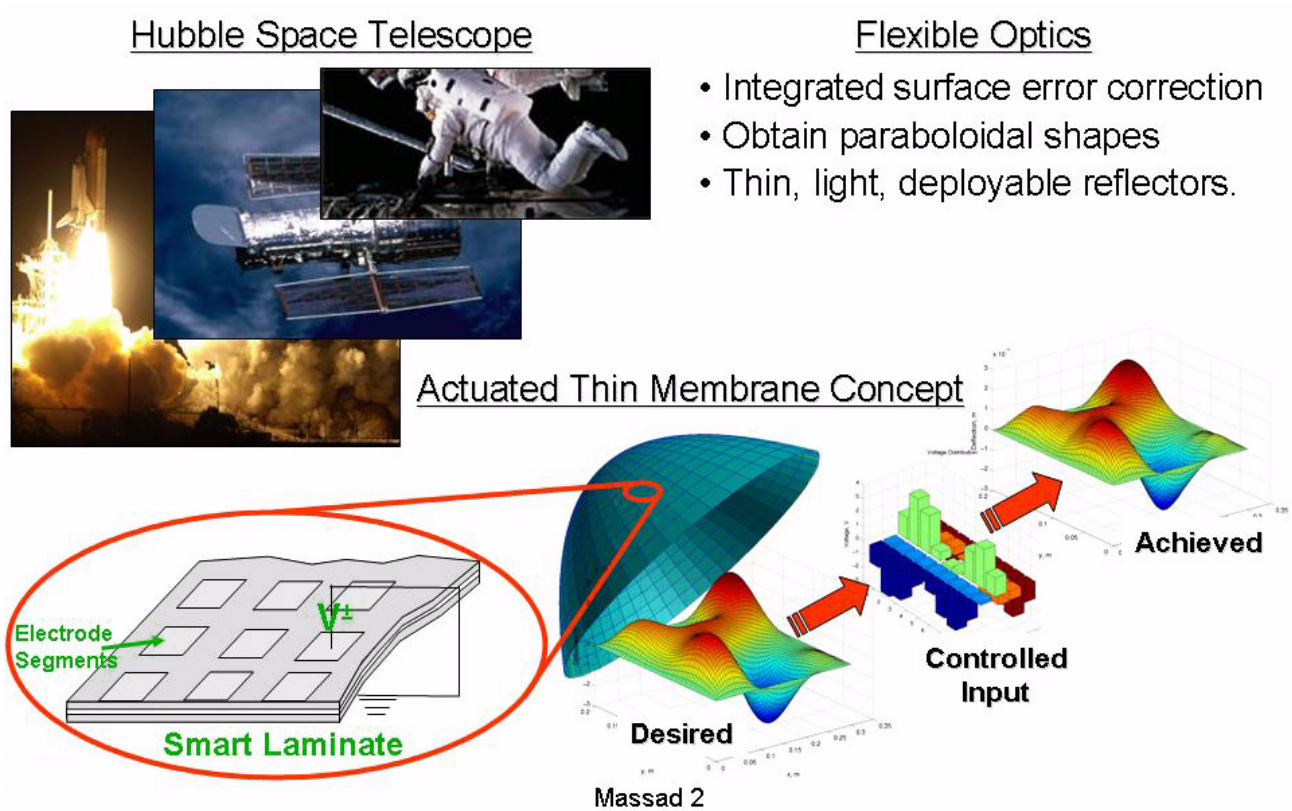
*67116*

*H. Sumali, J. E. Massad, P. L. Reu, J. W. Martin, P. M. Chaplya*


## Abstract

High resolution in antennas for monitoring the movement and proliferation of weapons of mass destruction demands large apertures. Current large aperture antennas are heavy and very expensive to launch. This project is developing a shape controlled deployable reflector that has the following features: (1) it is lightweight and membrane based, (2) it can be compactly folded or rolled before launch and then deployed on orbit, and (3) it is actively shape controlled to steer its beam. The shape control of flexible mirrors has been studied mainly for edge supported, thin plate configurations. For applications such as optical reflectors, corner supported configurations that allow for paraboloidal geometries prove more applicable. Moreover, the use of corner supports enables more flexibility and larger achievable deflections than the use of edge supports. This project focused on a shape controlled thin mirror consisting of an array of in-plane piezoelectric actuator segments laminated onto a corner supported substrate. An electric field distribution over the actuator segments causes the segments to expand or contract, thereby effecting plate deflection.

In FY 2004, we developed a conceptual design of the mirror material, packaging concept, and deployment idea. Our configuration of choice is a thin (about 100 micron) bimorph made of two sheets of piezoelectric polyvinylidene fluoride (PVDF). On the back side of this bimorph we etched an array of segmented electrodes. Application of a voltage over a segment results in deflection of the bimorph. To achieve a desired bimorph shape, we developed a theory for calculating the shape of the actuated reflector bimorph as a function of actuation voltage distribution. To validate our actuation theory, we investigated nonlinear finite element models of the smart laminate. In FY 2005, we


*Shape control concept.*

demonstrated that we can actuate a flat bimorph mirror into a paraboloid with a specified focus. We incorporated orthotropic material theory into our deflection model and developed a shape control algorithm that computes the voltage distribution that shapes the bimorph to the desired shape. We also demonstrated that we can shift the focus of the bimorph using the shape control algorithm, and we measured the shape of actuated reflector bimorphs.

## Accomplishments

First, we demonstrated that we can actuate a flat bimorph mirror into a paraboloid. Second, we developed an enhanced bimorph deflection theory to accommodate gaps between the actuators and orthotropic materials such as PVDF. This step also involved predicting the orientation of the orthotropic layers so that we can produce a circular paraboloid with a symmetric voltage distribution. Third, we developed a shape control algorithm that receives the desired shape and outputs the distribution of voltages to the actuator segments so that the bimorph deflects to the desired shape. Fourth, we developed a method to prescribe optical shapes in the interior circular aperture that smoothly satisfied our boundary conditions. Fifth, we demonstrated that we can shift the focus of the bimorph to vary its focus using Zernike polynomial analysis and the shape control algorithm. Sixth, we realized boundary conditions in a laboratory setup to approach the theoretical corner supported boundary condition. Seventh, we used advanced optical techniques for measuring the shape of the actuated reflector bimorph in the laboratory. Last, we developed advanced designs for next generation actuated bimorphs.

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# Micro Optical Radar (MOR) Facial Recognition Project

*73207*

*R. D. Habbit Jr., M. L. Koudelka, J. Jordan, R. R. Peters, J. V. Sandusky, D. J. Savignon, M. W. Koch, R. B. Taplin, R. O. Nellums, T. D. Russ, K. O. Wessendorf, C. Q. Little, A. D. Niese, S. K. Dunlap*

## Abstract

The purpose of this project is to improve security at US points of entry such as airports and borders, at critical US facilities, and to move the bad actor identification process abroad through the development of next generation three-dimensional (3-D) biometric identification. The fundamental enabling technology will be micro-optical radar (MOR) technology coupled with advanced recognition algorithms. We will focus on the use of facial recognition (FR) to address (1) the "9-11 Terrorist Security Problem" by autonomous identification of known terrorists and criminals at points of entry and critical facilities, and (2) the US security screening problem by helping to identify frequent "good actor" entrants to improve screening of unknowns and increase the flow of commerce. The recognition problem applies to many applications in which a person or automated process is attempting to identify an object from imagery taken from a distance, such as target recognition and intrusion detection. Recent news articles illustrated the poor performance of commercial 2-D systems and emphasized the difficultly in FR. We expect that the use of 3-D geometric facial images from MOR will improve performance to acceptable levels. Our approach requires two key technology advances: (1) a small image sensor to unobtrusively capture subcentimeter resolution 3-D images of moving subjects, and (2) new algorithm constructs to utilize the 3-D information.

In this project, the sensor development will specifically address the challenge of high resolution Flash MOR technologies at the single pixel level. It will also address the read-out challenges of the multipixel, complex, high speed, low noise, mixed mode MOR application specific integrated circuit. We will also examine the utility of 3-D information for FR and evaluate its sensitivity to certain parameters. This will enable 3-D FR sensitivities to be considered during the MOR research and development phase. We expect to demonstrate feasibility of Flash MOR and 3-D FR algorithms.

## Accomplishments

The primary goals were to understand the fundamentals of collecting 3-D data through Sandia sensor technologies and the fundamentals of 3-D data for recognition and identification through development of advanced efficient algorithms. Although many fields of use would benefit, this project focused on FR. We chose FR for two primary reasons: (1) its applicability to the critical national need for identification of persons in the War on Terrorism; and (2) its nonintrusive nature, ease of integration, and potential to identify individuals without subject cooperation. These attributes make FR ideal for screening at borders, airports, secure facilities, etc. World-wide 3-D FR continues to be the leading prospect for positive identification. FR also provides the broadest and greatest technical challenges.

The team completed an investigation into 3-D sensor technology, 3-D FR research, and the commercial companies offering such a capability. Since the MOR sensor technologies produce both 2-D and 3-D data, the team developed face segmentation approaches utilizing both data modalities. The combination of both modalities creates a powerful paradigm for recognition, especially when less than ideal 2-D imagery is available. Despite our success, the removal of hair regions is still an issue we need to investigate further. Perhaps tighter histogram thresholding on an already segmented face or further investigation of surface filtering techniques would lead to greater success in this area.

The team developed the two methods for facial feature extraction algorithms to provide an initial alignment for faces and to provide a basis for developing prescreening algorithms that achieve very high accuracy rates. We can locate the radial symmetry based features to within 10 mm of ground truth locations with 90% accuracy. While the multimodal features are less precise, we can locate them with an accuracy of 99.6% (by visual inspection) when good quality intensity images are available. The benefits of the multimodal approach become extremely apparent when the quality of either the 2-D or 3-D representation is compromised, as demonstrated in our robustness experiments.

We implemented and evaluated two main matching strategies: the 2-D range based Hausdorff algorithm and the iterative closest point algorithm. Computational costs led to the development of prescreening. The prescreener has a rank one recognition rate of 94%, a rank two recognition rate of 98%, and at rank six it has a 100% recognition rate. Prescreening the data is a valuable tool for 3-D FR systems. We developed a principal component analysis approach for modeling 3-D faces and demonstrated its utility for correcting facial expression variations. We conducted work to examine performance against a database with significant facial pose variations. The MOR sensor team successfully demonstrated feasibility at the single pixel level. The team also completed the design and fabrication of a 16 pixel array. The team continues to be very encouraged as all tests to date on the single pixel chips continue to demonstrate feasibility and improve in performance.

The technology research indicated that there are multiple hardware user groups. The original MOR sensor goal was a $10K or less cost in high volume applications such as airports. Initially, we knew that the MOR sensor concept would require significant nonrecurring engineering (NRE) costs, estimated at $40M or more. There is a low volume class of potential user groups, such as high value asset surveillance, that can tolerate a higher sensor cost but cannot support high NRE costs. For this latter application, the sensor team developed and successfully demonstrated a new laser radar sensor based on the use of an optical crystal. This sensor offers the potential to collect color 3-D images. The team successfully produced 3-D images with better than 0.250 inch resolution while operating at 1.5 microns.

## Refereed

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T. Russ, M. Koch, and C. Little, “3D Facial Recognition: A Quantitative Analysis,” presented and in *Proceedings of the IEEE Carnahan Conference on Security Technology*, Albuquerque, NM, October 2004, pp. 338–344.

M. Koudelka, M. Koch, and T. Russ, “A Prescreener for 3D Face Recognition Using Radial Symmetry and the Hausdorff Fraction,” to be published in *Proceedings of the IEEE Face-Recognition Grand Challenge Workshop at IEEE Computer Society International Conference on Computer Vision and Pattern Recognition*, San Diego, CA, June 2005.

T. Russ, M. Koch, and C. Little, “3D Facial Recognition a Quantitative Analysis,” presented and in *Proceedings of the INMM Annual Conference*, Phoenix, AZ, July 2005.

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## Other Communications

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# Adaptive Optical Zoom Sensor

*79842*

*D. V. Wick, B. Bagwell, T. N. Poland, W. C. Sweatt, B. F. Clark*

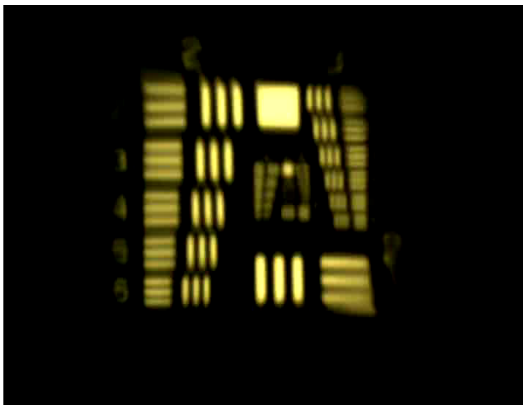
## Abstract

We based this project on the premise that there would be benefits to having a single imaging system that could quickly toggle between a wide field of view (FOV) for surveillance and situational awareness and a narrow FOV with higher resolution. In order to optically vary the magnification of an imaging system, continuous mechanical zoom lenses require multiple optical elements and use fine mechanical motion to precisely adjust the separations between individual or groups of lenses. The power requirements and slow speed of mechanical zoom lenses is prohibitive in many applications, including space based imaging.

By incorporating active elements into the optical design, we designed and demonstrated imaging systems that are capable of variable optical magnification with no macroscopic moving parts. We can change the effective focal length and magnification of an imaging system by adeptly positioning two or more active optics in the optical design and appropriately adjusting the optical power of those elements. In this application, the active optics (e.g., liquid crystal [LC] spatial light modulators or deformable mirrors) serve as variable focal length lenses.

Unfortunately, the range over which currently available active optics can operate (i.e., their dynamic range) is relatively small. Therefore, the key to this concept is to create large changes in the effective focal length of the system, with very small changes in the focal lengths of individual elements, by leveraging the optical power of conventional optical elements surrounding the active optics. By appropriately designing the optical system, these variable focal length lenses can provide the flexibility necessary to change the overall system focal length and, therefore, the magnification that is normally accomplished with mechanical motion in conventional zoom lenses.

We demonstrated both reflective and transmissive nonmechanical zoom systems. In addition, we developed a method for multispectral imaging using active optics to increase the spectral bandwidth. Finally, we quantitatively identified the benefits of using microelectromechanical system (MEMS) piston-tip-tilt mirrors as active optical elements.



*Video clip of zoom system with deformable mirrors.*

## Accomplishments

We successfully demonstrated a 4x (i.e., an increase in magnification of 4) all reflective active optical zoom system with a 2x increase in resolution capability. Using micromachined deformable membrane mirrors (MMDMs), we demonstrated a system with no macroscopic moving parts that could toggle between wide and narrow FOV in less than 10 milliseconds with near diffraction-limited performance. We also demonstrated a 2x all refractive system that used LC lenses as the active optical elements. The main limitation to the concept for both reflective and refractive systems is the relatively small dynamic range of currently available active optical elements. Therefore, given current technology, the most promising implementation of nonmechanical zoom appears to be space based or near space imaging. In these cases, the size of the imaging system can be fairly large, which lends itself to the larger optical paths that are required for nonmechanical zoom.

While MMDMs were useful for a proof-of-principal demonstration, robustness and resonance issues make them a poor choice for fieldable systems. MEMS segmented mirrors offer a more robust alternative. In addition, MEMS mirrors are not electrostatically driven, which means that they do not necessarily impart defocus when they are actuated, as MMDMs do. This increases their functionality, as other aberrations (i.e., tilt, astigmatism, coma, or spherical aberration) can be imparted by the MEMS device without adding

unwanted focus. Sandia is currently developing optical quality MEMS mirrors with 85 individual elements. Each element has piston, tip, and tilt capability and 27 microns of stroke.

Adding tip and tilt capability to the MEMS mirrors greatly reduces the total number of elements we need for imparting a given wavefront. As part of this effort, we performed a thorough analysis comparing piston-only and piston-tip-tilt for segmented MEMS mirrors. The results of this study showed that piston-tip-tilt can significantly improve the performance of these devices.

The LC lenses that were available, both commercial and prototype devices, were not capable of diffraction limited performance. However, we were able to demonstrate zoom capability using both, showing that the concept is viable. Unfortunately, LC lenses tend to add spherical aberration when defocus is applied. However, we found that new prototype devices from Holochip, Inc., are much better than other available LC lenses. In addition, these devices are promising because they can potentially be cascaded to increase the dynamic range and hence reduce the size of the optical system. Unfortunately, they are inherently slow and take several seconds to reach equilibrium. We are working with Holochip to address this issue.

Finally, we developed a concept for increasing the spectral bandwidth of an imaging system by integrating a variable focal length optic with a nonmechanical spectral filter. Although not directly related to active optical zoom, this idea may turn out to be extremely useful for multispectral imaging in some applications. Due to the elimination of axial chromatic aberrations by simply adding defocus, we could use a single optical system to image a wide range (i.e., 400–1700 nm) by simply looking at a single wavelength at any given instant. Thus, by incorporating a variable focal length active element and a nonmechanical spectral filter, a single multispectral imaging sensor could toggle between wavelengths at near kHz speeds.

## Refereed

W.C. Sweatt, “Reduction of Zernike Wavefront Errors Using a Micromirror Array,” to be published in *Optical Engineering.*

## Other Communications

D.V. Wick, D.M. Payne, T. Martinez, and S.R. Restaino, “Large Dynamic Range Wavefront Control of Micromachined Deformable Membrane Mirrors,” presented and in *Proceedings of the SPIE Defense and Security Symposium*, Orlando, FL, March 2005, vol. 5798, pp. 158–161.

D.V. Wick, T. Martinez, D.M. Payne, W.C. Sweatt, and S.R. Restaino, “Active Optical Zoom System,” presented and in *Proceedings of the SPIE Defense and Security Symposium*, Orlando, FL, March 2005, vol. 5798, pp. 151–157.

T. Martinez, D.V. Wick, D.M. Payne, and S.R. Restaino, “Active Optical Zoom for Laser Communication,” presented and in *Proceedings of the SPIE Defense and Security Symposium*, Orlando, FL, March 2005, vol. 5793, pp. 144–147.

# Compensation of Ionospheric Errors for Geolocation

*79843*

*J. J. Mason, A. Romero, C. L. Leger, R. M. Holman, R. Connell, D. D. Kiffer, C. R. Collins, R. M. Axline, L. Romero*

## Abstract

Satellite based geolocation systems play a crucial role in public safety and national security missions in accurately locating objects on Earth. This project addresses the ionospheric effects that limit the accuracy of current radio frequency (RF) satellite based geolocation systems. Although techniques for ionospheric correction for time of arrival (TOA) systems are common, we are trying to establish similar techniques for frequency of arrival (FOA) and phase of arrival (POA) systems.

In order to improve the accuracy of satellite based TOA and FOA based geolocation systems, we wish to correct the effects the ionosphere has on RF signals. These effects include small time delays due to ionosphere total electron content (TEC), and small frequency or Doppler shifts due to ionospheric TEC rate of change. We are attempting to experimentally measure the size of the ionosphere errors and to determine whether these errors can be predicted, and compensated for in real time.

To compensate for FOA errors, we are investigating estimation of the TEC rate in near real time from operations on real time TEC data obtained from a particular source. A second method of compensating for ionospheric errors is cancellation using measurement differencing techniques. We propose differencing POA measurements to potentially yield a novel and more accurate method of geolocation. We believe we have identified a way of dealing with the cycle ambiguities and sources of phase error that previously prevented the use of phase in our systems.

## Accomplishments

We set up a ground station to measure the ionospheric TEC and TEC rate using existing Sandia satellite assets. The ground station consists of a transmitter and three receivers. We erected three 14 foot reflector type antennas to feed the three receivers. We integrated an antenna control system with each reflector to enable the tracking of satellites. We installed a global positioning system (GPS) based time and frequency standard to supply all other components, which must operate synchronously, with reference clock signals. The reflector outputs are digitized for computer processing. We wrote control software to coordinate the continuous automated operation of the transmitter, receivers, digitizers, and subsequent signal processing. We wrote data processing software for tasks including signal detection, up-link and down-link Doppler correction, RF link level prediction, spectral analysis, and troposphere and ionosphere prediction.

We also purchased a GPS wide area augmentation system (WAAS) receiver, and wrote software to extract and record real time ionospheric TEC data from the WAAS messages. The recorded TEC data are stored, thereby creating a database from which we can compute the TEC and TEC rate for any location and time/date. We implemented a Chebyshev polynomial based algorithm to differentiate the noisy WAAS TEC data to give TEC rate data, and are evaluating this algorithm.

A major goal of the project was to compare the TEC rate measurements to the WAAS derived TEC rate data. We hoped to validate the concept of producing TEC rate estimates by a combination of fitting/smoothing and differentiating TEC data. We are presently attempting to make this comparison, but the ionosphere TEC rate is small lately, as we are in a period of low solar activity. In fact, present TEC rates are just below our measurement ability, so we are currently trying to reduce our measurement errors. Our dominant source of error appears to be our station reference oscillator. This oscillator is GPS disciplined and is achieving a respectable accuracy of 0.1 Hz in 2 GHz (5E-11 fractional error). The WAAS data indicate current ionosphere errors here in Albuquerque are more like 0.01 Hz. So while we are not currently achieving this goal, we continue to work on it.

We are beginning to work on our second goal of demonstrating a phase difference of arrival (PDOA) geolocation scheme. We began by geolocating our own continuous wave (CW) transmitter, as well as several other CW signal sources of opportunity, using an instantaneous FOA technique recently developed at Sandia. While it is likely that others have explored similar techniques, instantaneous FOA geolocation (as opposed to the widely used Doppler-profile technique) does not yet appear in the open literature.

## Refereed

J.J. Mason and L.A. Romero, "TOA/FOA Geolocation Solutions Using Multivariate Resultants," to be published in *NAVIGATION, The Journal of the Institute of Navigation.*

# Single Photon Sensitive Imaging Detector Arrays

*79844*

*M. S. Carroll, D. K. Serkland, J. F. Klem, K. M. Geib, I. J. Fritz, R. K. Patel, J. L. Rienstra*

## Abstract

We propose to develop avalanche photodiodes (APDs) with low dark counts (< 4.5E12 events/(sec·cm$^2$)), high multiplication (> 300), and high quantum efficiency at 1550 nm (> 10%) for single photon sensitive imaging detector arrays. This will constitute a new optoelectronic device capability that can scale to fairly large imaging arrays (256 x 256), given sufficient investment. The key need is short wave infrared light detection and ranging (LIDAR) imaging at temperatures greater than 100 K.

A primary challenge to achieving high sensitivity APDs at 1550 nm is that the small bandgap materials (e.g., InGaAs or Ge) necessary to detect low energy photons exhibit higher dark counts (due to tunneling, defect assisted currents, or diffusion currents) and higher multiplication noise (due to nonideal ratios of electron and hole ionization coefficients) compared to materials like silicon.

To overcome these historical performance problems, we will design and fabricate APDs using separate absorption and multiplication (SAM) regions to leverage the 1550 nm sensitivity of either InGaAs or Ge. This will allow us to achieve silicon-like multiplication properties in Si, SiGe, or heterostructure engineered InAlAs multiplication regions. The novel integration of narrow and wide bandgap materials promises to be a significant scientific challenge that will expand our capabilities.

## Accomplishments

We proposed the following milestones to develop single photon imaging detectors: (1) design of APDs using available literature data and numerical simulation; (2) establishment of germanium material sources including introduction of a germanium process; and (3) development of a baseline fabrication of 1550 nm sensitive APDs using InGaAs/InAlAs, Ge/Si, SiGe/Ge designs. We fabricated InGaAs/InAlAs APDs, and we are currently resolving integration issues for the Ge/Si devices.

Specifically, we completed designs of APDs using the available literature data combined with numeric simulation. This resulted in designs for: (1) Geiger mode operation to overcome the difficulties of gain noise in germanium and InGaAs compatible materials, and (2) analog detection with multiplication gain in a silicon region that includes a novel approach to ameliorate the noise producing material defects in lattice mismatched germanium on silicon. We evaluated and established germanium sources in order to fabricate the Ge/Si and SiGe/Ge detectors. We also introduced a new germanium deposition capability. We completed masks and a proposed process flow, which necessitated the development of new germanium fabrication processes (e.g., new etches and compatible dielectric deposition).

Furthermore, we epitaxially grew baseline InGaAs-InAlAs devices; fabrication and measurements are in progress. Initial direct current electrical measurements are similar to state-of-the-art performance published in the literature. Finally, we constructed critical measurement setups to evaluate material and device quality. This includes: (1) a new measurement technique to determine bulk minority carrier lifetimes, a critical material property for detectors; and (2) a low temperature gated Geiger mode measurement setup to evaluate single photon operation of these APDs, such as dark counts and photon detection efficiency. This enabled the demonstration of Geiger mode operation at 16K in the Sandia InGaAs-InAlAs APD. Evaluations of the Sandia APDs' higher temperature Geiger mode characteristics are in progress.

## Refereed

J.J. Sheng and M.S. Carroll, "Minority Carrier Lifetime Measurement of Recrystallized Amorphous Germanium and Poly-Crystalline Germanium on Silicon for Optoelectronic Applications," Pentagon Report, Materials Research Symposium, Warrendale, PA, December 2005.

# Optimal Spectral Unmixing of Remotely Sensed Hyperspectral Images Without A Priori Knowledge of the Endmembers

*79845*

*C. L. Stork, M. R. Keenan*

## Abstract

The International Atomic Energy Agency (IAEA) and other nuclear regulatory agencies have considerable interest in utilizing satellite remote sensing for the identification of undeclared uranium mining and milling activities and the verification of declared uranium mining and milling. However, a systematic evaluation of the utility of satellite remote sensing for differentiating uranium mining and milling operations from other types of mineral mining and milling operations has not been performed. We address this deficiency and systematically evaluate the ability of current generation, satellite based spectroscopic sensors to distinguish uranium mines and mills from other types of mineral mining and milling operations.

We perform this systematic evaluation by (1) outlining the remote, spectroscopic signal generation process, (2) documenting the capabilities of current commercial satellite systems, (3) systematically comparing the uranium mining and milling process to other types of mineral mining and milling operations, and (4) identifying the most promising observables associated with uranium mining and milling that can be identified using satellite remote sensing. It is important to outline the remote spectroscopic signal generation process in order to understand which portions of the electromagnetic spectrum we can use to remotely monitor mining and milling activity.

Documenting the capabilities of current commercial satellite systems is important to understand spectral and spatial resolution limitations to mining and milling monitoring. Performing a systematic comparison of uranium mining and milling to other types of mineral mining and milling operations is key in identifying observables unique to, or characteristic of, uranium mining and milling. Based on this comparison, we identify the most promising observables associated with uranium mining and milling that can be exploited remotely. We use the Ranger uranium mine and mill in Australia, which is a well known site that has a large body of open literature information, to illustrate the systematic approach we are proposing.

## Accomplishments

Based on our systematic review of the mining and milling literature, we determined that differentiating uranium mining and milling operations from other types of mineral mines and mills is a difficult task. This is attributable to the fact that uranium mines and mills share many common processes and potential observables with other types of mineral mines and mills. Due to the limited number of unique, potential observables associated with uranium mining and milling, it may be necessary to utilize multiple, nonunique observables to reach a conclusion regarding the type of mineral extraction at a mining and milling site. The information contained in multiple, nonunique observables can be used to narrow down the number of possible mineral mining and milling activities carried out at a particular site, and potentially even unambiguously identify the type of mineral extracted at that site.

Based on our literature research of uranium mining and milling, and research of mining and milling practices used for other types of minerals, we developed a decision tree that utilizes the information contained in one or more observables to determine whether uranium is possibly being mined and/or milled at a given site. Potential observables associated with uranium mining and milling included in the decision tree are: uranium ore (typically less than 1 percent uranium by volume), the radiometric discriminator station (used for uranium ore sorting), sulfur (used to produce sulfuric acid), sulfuric acid (used in leaching the uranium ore), manganese dioxide (used as an oxidant in leaching), sodium chlorate (leaching oxidant), the uranium pregnant leach liquor (contains uranyl sulfate), ammonia (used in precipitation of yellowcake), and uranyl compounds disposed in the tailings pond (24 ppm uranium).

Few of the observables included in the decision tree can be identified spectrally using current satellite sensors. Hyperion, which is the only commercially available hyperspectral satellite system, offers high spectral resolution (220 bands from 0.4 to 2.5 microns), but limited spatial resolution (30 m ground resolution). At the Ranger site in Australia, the open pit mine containing the uranium ore and the tailings pond contain-

ing uranyl waste products are very large and can be seen in Hyperion images. However, uranium cannot be detected in the open pit mine and in the tailings pond using Hyperion because it is present at very low concentrations. Several smaller, potential observables at Ranger, such as the radiometric discriminator station and the ammonia tanks, are not visible in Hyperion images due to the sensor's 30 m spatial resolution. However, we can see and spectrally identify the sulfur stockpile at Ranger using Hyperion. The sulfur stockpile is used at Ranger to produce sulfuric acid, which is required for uranium ore leaching. Employing Hyperion, the remote identification of sulfur indicates that acid leaching is used at the Ranger site. Consulting the decision tree, the fact that acid leaching is used at the Ranger site narrows the number of possible mineral candidates to uranium, copper, zinc, manganese, vanadium, the rare earths, and phosphorus.

## Other Communications

B. Jasani, H.A. Smartt, D. Blair, C.L. Stork, J. Smith, and M. Canty, "Evaluation of Remote Sensor Systems for Monitoring Uranium Mines," to be published in *Proceedings of the European Safeguards Research and Development Association 27th Annual Meeting.*

# Fully Integrated Microfluidic Microthruster System for Micropropulsion Applications

*79846*

*K. D. Patel, K. Wally, M. C. Grubelich, M. L. Decker, K. A. Peterson*

## Abstract

Nanosatellite system constraints on mass, volume, power, and fuel pose several challenges to designing an efficient micropropulsion system for orbit control and high altitude maneuvers. We propose to build a fully integrated micropropulsion system as an enabling technology for nanosatellite systems. The design is based on electrokinetic (EK) pumping of liquid propellants in a novel extreme temperature and pressure capable ceramic microfluidic device. Voltage controlled EK pumping is an elegant method for metering propellants from the nL/min to mL/min flow rates to achieve low micronewtons (attitude control) to millinewtons (vector thrusting) of force. We fabricated the microthruster housing from thin sheets of ceramic that are laser cut, stacked, and bonded to form a sintered one piece device that incorporates fluid microchannels, a propellant mixer, a thrust chamber, and an exhaust nozzle.

Common rocket propellants such as hydrogen peroxide and hydrazine have properties amenable for EK pumping; however, such fluids have never been tested. We plan to evaluate and characterize the EK pumping performance of concentrated hydrogen peroxide as a monopropellant for thrust generation and efficiency. Subsequently, we plan to test more energetic propellants (hydrazine) with an oxidizer (hydrogen peroxide) for use in a hypergolic bipropellant microthruster for greater thrust efficiencies.

Direct EK pumping of propellants requires no moving parts, and we can fashion EK pumps in different configurations for increased flexibility in designing a monopropellant and/or bipropellant system for a nanosatellite system. The high fluid impedance of the EK pump to pressure driven flow means no valving is necessary, which simplifies the microthruster to a small lightweight package. Fabricating the housing in ceramic offers a straightforward and efficient strategy for micropropulsion systems in satellites for this weight class.

## Accomplishments

We fabricated voltage controlled EK pumps with no moving parts in fused-silica capillaries by high pressure slurry packing 0.5 micron spherical silica particles. We achieved a significant milestone when we experimentally demonstrated silica EK pumps to directly pump anhydrous hydrazine. We obtained performance efficiencies of 6 psi/volt and generated pressures as high as 3000 psi. We also directly pumped hydrogen peroxide using a surface modified EK pump; we measured performance efficiencies of 3 psi/volt. We developed a new high pressure gasless electrode. We tested the electrode design to 5000 psi with an EK pump, and it exhibited no bubble generation. We investigated both hydrazine and hydrogen peroxide propellants in a monopropellant and bipropellant configuration. Using microforming techniques, we formed thrust chambers and exit nozzles in fused silica capillaries to determine the optimal catalyst for a monopropellant configuration. We achieved preliminary microthrusting in monopropellant and bipropellant configurations. We accomplished the initial testing of a ceramic microfluidic thruster with a simple three port design. We are currently developing future ceramic designs to optimize performance based upon these observations.

## Other Communications

K.D. Patel, K.A. Peterson, and R.W. Crocker, “Microfluidic-Based Microthrusters for Nanosatellite Propulsion,” presented and in *Proceedings of the Gordon Research Conference, The Chemistry and Physics of Microfluidics*, Oxford, England, August 2005, CD-ROM.

# Standoff Detection of Explosives Using UV Lidar Technology

*79847*

*P. J. Hargis Jr., L. R. Thorne, J. E. Parmeter, C. C. Phifer, R. L. Schmitt, D. W. Hannum, C. C. Hayden, M. A. Gutzler*

## Abstract

Terrorist acts in recent years, including the successful employment of vehicle bombs against US targets, have led to a heightened need for technologies for explosives detection. Current detection techniques are local in nature, and detection at substantial standoff distances represents a huge technical challenge. Vapor plumes emanating from explosive charges are generally too dilute, and probing bulk techniques involving x-rays or neutrons suffer from an intrinsic $1/r^4$ decrease in signal intensity with increasing standoff distance. At present, detection is very difficult even at a standoff distance of 10 meters. This project investigates the use of ultraviolet (UV) lidar as a technique for standoff explosives detection. The focus is on the use of laser induced fluorescence to detect explosives and impurities contained in explosive particulates (a few hundred nanometers to a few microns in diameter) adsorbed on surfaces.

Previous studies of contamination on vehicles loaded with explosives indicate that such particulates are likely to be present on the surface of such a vehicle. Therefore, this technique could prove useful for the standoff detection of large vehicle bombs. The approach proposed here differs from previous studies in that the primary target is explosive particulates rather than vapor. A primary goal is to demonstrate standoff detection of at least one key explosive (ammonium nitrate, TNT, or a plastic explosive based on RDX or PETN) at a distance of 10 meters or greater.

## Accomplishments

We focused on laboratory studies of the fluorescence properties of explosive particulates adsorbed on a variety of surfaces. The explosives were of two main types, nitro based explosives and explosives based on ammonium nitrate. The nitro based explosives were 2,4,6-trinitrotoluene (TNT), cyclonite (RDX), octogen (HMX), and pentaerythritol tetranitrate (PETN). The explosives based on ammonium nitrate included both ammonium nitrate (AN) and ammonium nitrate soaked with fuel oil (ANFO). We observed fundamentally different results for these two categories of materials. We observed virtually no intrinsic fluorescence from particulates of TNT, RDX, HMX, or PETN, but adsorption of these particulates onto a highly fluorescing $MgF_2$-coated gold surface (protected gold, or PG) led to a dramatic reduction in the fluorescence of the surface. We saw this quenching effect at excitation wavelengths of both 355 and 266 nm, and it occurred with both prompt and delayed fluorescence.

The signal was reduced by at least an order of magnitude from the maximum intensity, which suggests the possibility of detecting nitro based explosive particulates on surfaces by monitoring the quenching and/or polarization properties of the fluorescence associated with the surface. We applied the nitro based explosives to painted surfaces to test the generality of this phenomenon. Although quenching also occurred on the painted surfaces, the effect was not as dramatic as that seen on PG.

In contrast to the nitro based explosives, ANFO particulates do exhibit their own inherent fluorescence. Pure AN exhibits virtually no fluorescence at the excitation wavelengths studied. However, commercial explosive grade, low density ammonium nitrates do fluoresce because of substances such as organic surfactants that are added during the manufacturing process. The surfaces of the AN prills must be treated to prevent caking that would otherwise occur because of the hygroscopic nature of ammonium nitrate.

The oil in ANFO is usually #2 diesel fuel. Of the over 400 components in diesel fuel, we only expect a few (the polycyclic aromatic compounds) to fluoresce over the wavelength range we investigated. The fluorescent species in diesel fuel yield strong signals, but their lifetimes are fairly short. At the excitation wavelength of 355 nm, diesel fuel shows no detectable fluorescence if a delay of 1 microsecond is employed.

We began an attempt to determine whether ANFO spectra have a fluorescence signature that differs from that of either AN or diesel fuel alone. We discovered that the fuel soaked AN prills do show an unusual feature. It appears that the diesel fuel fluorescence lifetime is prolonged when the emitting molecules are adsorbed on the surface, as compared to the lifetime associated

with diesel fuel in its liquid state. This means that a diesel like spectrum can be seen at a delay of 1 microsecond in ANFO. At the same delay time, diesel fuel alone shows no signal, and the ammonium nitrate spectrum occurs in a different spectral region. Therefore, we can distinguish ANFO from either of its ingredients. These results could potentially lead to a method for standoff detection of ANFO.

# Fail-Safe Infectious Substance Transport Packages

*79848*

*M. D. Tucker, J. M. Gaudioso, B. L. Levin, C. A. Souza*

## Abstract

As President George W. Bush recognized in November 2001, "Infectious diseases make no distinctions among people and recognize no borders." By their very nature, infectious diseases of intentional (bioterrorist) or natural origins have the ability to threaten the people and economics throughout a region. The best mechanism for minimizing the spread and impact of infectious disease is rapid diagnosis. Rapid diagnosis enables decision makers to determine the best response mechanism to contain the spread of the disease. However, for rapid diagnosis to occur, infectious substances (ISs) must be transported to appropriate laboratories sometimes located across the world. Shipment of ISs, including those resulting from a bioterrorism event, is problematic since many carriers concerned about leaking packages refuse to ship these materials. Air carriers are especially concerned since leakage of ISs may require a difficult and costly decontamination of their aircraft. Current packaging for the shipment of ISs does not have any ability to neutralize or kill leaking ISs.

The objective of this project is to develop a safe and secure packaging method (i.e., a "fail-safe" packaging method) for the transport of ISs 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. The packaging method will contain a novel decontaminating material that will kill or neutralize any infectious organisms that leak from the primary container. This self-decontaminating feature of the proposed packaging method will alleviate concerns of ground based or air based carriers, and will allow lower risk shipment of infectious substances.

## Accomplishments

Our work focused on three tasks: (1) incorporation of the Sandia DF-200 formulation into absorbent material (Task 1), (2) preliminary efficacy testing (Task 2), and (3) stakeholder interactions (Task 3).

In Task 1, we developed a material capable of both absorbing and neutralizing infectious organisms in an IS package. This material incorporates a super absorbent gel (amorphous silica) and the fundamental ingredients of the Sandia Decon Formulation in solid form. A primary consideration was the development of an absorbent/neutralization material with a high stability. The Sandia technology is based on a low concentration of hydrogen peroxide that works in synergy with other ingredients. However, hydrogen peroxide can slowly degrade (especially at elevated temperatures). Therefore, a primary focus of this task was to identify a form of solid hydrogen peroxide with high stability at elevated temperatures. We evaluated the various forms of unencapsulated and encapsulated solid peroxide compounds and selected the best compound for this application to minimize degradation.

In Task 2, we conducted a series of tests to optimize the ratio of absorbent/neutralization material to volume of IS. We also conducted efficacy tests against the various types of pathogenic organisms in pure culture solutions and in solutions with a heavy organic loading to mimic bodily fluids or environmental samples that contain ISs.

In Task 3, team members met with potential stakeholders from both the United Nations World Health Organization and the US Department of Transportation. These meetings helped us establish the design criteria for a prototype, including the types of specimens the package would need to accommodate. This information will enable the project team to design a prototype that will be compliant with current and future regulations.

# Development of Entomologic Surveillance to Aid Early Disease Detection (EDD)

*79849*

*S. A. Caskey, M. Aspelin, M. P. Wilson, H. A. Smartt*

## Abstract

Insects can and have been used as bioweapons since the 1800s. Entomological surveillance would provide important data in a first response to such a bioweapon attack, aid in the tracking of emerging diseases, and could be used as a tool to promote regional stability.

In the event of a bioweapon or an epidemic onset, experts predict that the insect population would carry detectable traces of the disease well before humans or animals show any symptoms. Insects can also serve as potential bioweapon vectors by carrying and transmitting a bioweapon disease or by introduction into an area where they could be agriculturally and economically devastating as agricultural pests. All of these are cross border concerns and can often be the subject of heated political arguments.

Early disease detection works to reduce the spread of insect borne disease, whether naturally occurring or introduced, by developing models to aid in identifying and predicting where insects are, patterns of migration, and estimation of insect population densities. We are developing the models using remote sensing data in combination with ground collected insect samples.

With the use of remote sensing and geographic information systems (GISs), it may be possible to predict where insects are currently located and where they may migrate to in the future. Analysis of satellite imagery, when tied to weather patterns, soil content, and elevation, may lead to the ability to track disease carrying insects both within a country and across borders.

## Accomplishments

We focused the initial development of the insect tracking model to a limited set of species; these are the species that carry and transmit the West Nile virus (WNV) and eastern equine encephalitis (EEE). Of increasing concern within the US is WNV, introduced via a mosquito vector. EEE is one of the US select agents, which are pathogens or toxins considered of greatest risk to the US and possible bioweapons agents.

These vector species are in existence in the northern US, including Connecticut, which is the location of Yale University's vector ecology laboratory. This laboratory is working in collaboration with us on this project. They have extensive knowledge of insect disease vectors and some experience with remote sensing and GISs. We tasked the researchers at Yale University to develop a sampling methodology and provide the geographic coordinates of the sampling areas to help define the swath sizes for the remote sensing data. They collected mosquito samples within the sampling areas during the summer of 2005. They are also responsible for abundance estimates and providing the details of each habitat and sampling site.

We tested the Sandia managed multispectral thermal imager (MTI) and the USGS EO-1 Hyperion and ALI satellites and began analysis of imagery over the identified urban and rural swaths. We collected ground data to aid in analysis and will continue to collect it as needed. The ground data come in the form of mosquito trap data and larvae collections from the soil. We will investigate one urban and one rural swath in order to determine the differences between the habitats and mosquito behaviors. The urban/rural relationship has never been investigated and will provide insight into ways of handling bioweapon introduction in the different habitats.

# Biological Risk Assessment Methodology (BioRAM)

*79850*

*J. M. Gaudioso, R. M. Salerno*

## Abstract

There is no widely accepted methodology for determining the appropriate protection level for biological facilities that store and use dangerous biological agents. We are developing a risk assessment methodology for biological facilities (BioRAM) to help determine appropriate protection strategies for dangerous pathogens and toxins. We focused on developing a biosecurity program for implementation at these facilities based on the biological assets they hold. The process includes an evaluation of the biological agents' consequences of use and weaponization potential, the facility environment, and the characteristics of potential adversaries.

The project includes the following tasks:

(1) Constructing a methodology for prioritizing biological agents based on weaponization attributes (acquisition, production, and dissemination) and consequence attributes (transmissibility, morbidity, mortality, psychological impact, and economic impacts).

(2) Building a facility risk assessment model that includes the biological agents, other assets (e.g., research animals and facilities), and the threat environment. Evaluation of the threat environment includes a systematic method for analyzing the means, motive, and opportunity of potential adversaries.

(3) Developing a computer tool for conducting facility security risk assessments. We will design this tool for use by management, or those supporting management, to help evaluate the risks to the facility's assets. It will demonstrate the magnitude of risk associated with theft and use of their biological assets as weapons, as well as theft or destruction of their physical assets. The tool will recommend which risks to address with security measures. The tool will also recommend a biosecurity program based upon the facility risks. Three possible configurations included are for facilities: (1) with moderate risk assets; (2) with high and moderate risk assets; or (3) with very high, high, and moderate risk assets. We will also document vulnerabilities that exist between the current state of security and the recommended biosecurity program in order to facilitate upgrade and reassessment activities.

## Accomplishments

We focused on three tasks.

- *Task 1: Technical analysis of candidate biological weapon agents:* We developed a framework for evaluating the malicious use risk of agents. We established the attributes that form the basis for this; they have been peer-reviewed. We finalized the value tables that allow for a quantitative evaluation and uncertainty analysis of agent risk. We populated a database of evaluated agents for use in the model. We added data on 30 agents from a Department of Homeland Security funded project to the database. We evaluated the full spectrum of biological toxins that are often considered to be potential bioterrorism agents for the database. In order to keep this as an unclassified tool usable at overseas bioscience facilities, we limited the agent characteristic analysis to what is available through the open literature.
- *Task 2: Preliminary algorithm development and validation:* We successfully formalized our methodology for biological facility risk assessment. We defined attributes for adversary potential (motive, means, and opportunity) and drafted the values of various risk mitigation techniques to address insiders, outsiders, and the level of risk they represent. We integrated the agent specific attributes from Task 1 into the overall BioRAM methodology. External and Sandia internal experts participated in establishing the appropriate values assigned to the different elements of the threat assessment.
- *Task 3: Validation by risk assessment community:* We presented the integrated BioRAM model for critique and validation to a group of Sandia security and risk assessment managers.

## Refereed

N. Barnett, J. Gaudioso, and R.M. Salerno, "Security Risk Assessment Methodology for Bioscience Facilities," presented and in *Proceedings of Working Together: R&D Partnerships in Homeland Security Conference*, Boston, MA, April 2005, p. 12.

## Other Communications

J. Gaudioso, R.M. Salerno, and N. Barnett, "Developing a Risk Assessment and Management Approach to Laboratory Biosecurity," to be published in *Applied Biosafety.*

# Extreme Response Composite Materials for Sensor Applications

*79851*

*J. E. Martin, R. L. Williamson, R. A. Anderson*

## Abstract

Field structured composites (FSCs) are a new class of patented Sandia materials that show great potential as conduction based sensors. We synthesized FSCs by suspending conducting magnetic particles in a polymeric resin and subjecting them to structuring magnetic fields during cure. The particle assemblies, such as chains, conduct current, but the one-dimensional conduction paths are sensitive to swelling or deformation of the polymer. In preliminary studies, we demonstrated reversible piezoresistance of 11 decades, reversible thermoresistance of 10 decades, reversible chemiresistance of 6 decades with 50 ppm xylene, and magnetoresistance of 4 decades in an induction field of 1200 gauss. The conductor-insulator transition temperature is tailorable, and the magnetoresistance could offer a method of dynamically controlling response.

Two additional factors make these robust materials attractive for sensing. First, FSCs lend themselves to dual-input sensing. Near the conductor-insulator transition, the conductivity is sensitive to magnetic fields, stress, pressure, and chemical vapors. Second, the structure of the particle assemblies can be controlled over a wide range due to the discovery that three-dimensional magnetic fields can be used to create a variety of complex particle assemblies, such as sheets and honeycombs. These structures should enable us to tailor the FSC response.

We propose a detailed investigation of the response of FSCs to single and dual inputs, including temperature, pressure, strain, magnetic fields, and chemical vapors. We will conduct these measurements on a variety of composite structures synthesized in our resonant triaxial Helmholtz coil using both glassy and rubbery polymers.

## Accomplishments

We set up three systems for the evaluation of chemical sensors: (1) a flow system that enables the determination of chemiresistor response (resistance change), (2) a quartz crystal balance for measuring mass uptake of polymer thin films, and (3) a microbalance for measuring mass uptake in polymer thin films and in the chemiresistors themselves. With this equipment, we can relate the resistance change to the degree of swelling. Using these tools, and various magnetic field processing, we greatly increased the sensitivity of our sensors, which can now detect as little as 300 ppb toluene. We achieved full conductor-insulator switching at only 4 ppm toluene. We also demonstrated that the application of modest (1 kilogauss) magnetic fields can alter chemiresistor response by a factor of 120,000! (At the same fields, extreme magnetoresistance is a factor of 1.5.) We also demonstrated that it is possible to sense volatile organics by changes in the light output of electroluminescent phosphors (ZnS), and completed an investigation of the decay dynamics of these sensor materials. We also showed that it is possible to create a material that spontaneously switches from an insulator to a conductor after several weeks, and that this switching time is tunable. We used triaxial magnetic fields to create a sensor with network-like particle agglomerates and showed that this gives improved sensitivity and magnetic field switching.

# Laser Induced Breakdown Spectroscopy for Remote Explosives Detection

*79852*

*L. A. Theisen, L. R. Thorne, S. M. Sickafoose*

## Abstract

We are investigating the feasibility of using laser induced breakdown spectroscopy (LIBS) in combination with sample preconcentration for the remote detection of explosives. Our emphasis is on the detection of large vehicle bombs, for which there is currently no remote detection capability. Developing a successful capability would save lives by increasing the distance between the explosives and the intended targets and/or security forces. We completed the first of a multiyear study to evaluate the use of a rugged LIBS spectrometer suitable for use on a mobile robotic platform to detect explosives.

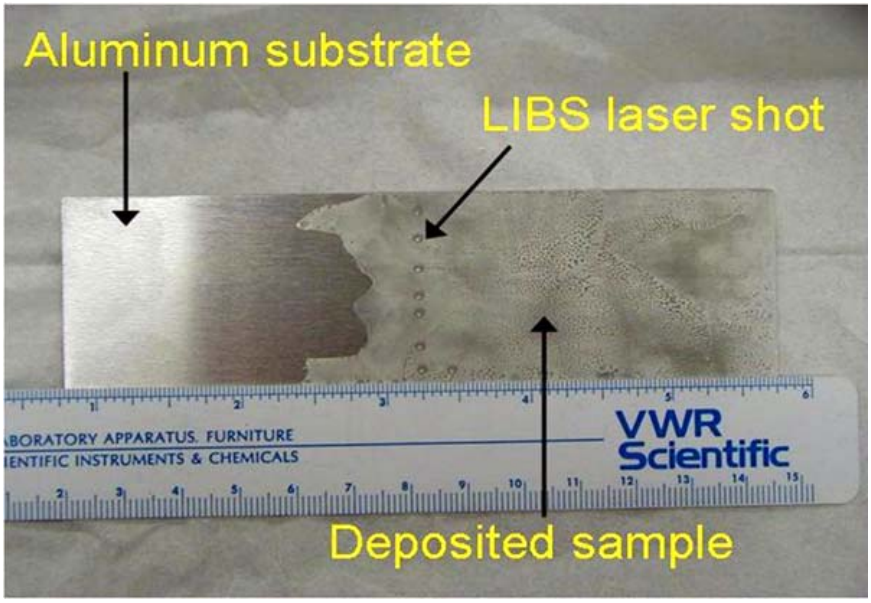
Specifically, this study involves evaluating the collection, preconcentration, and detection of explosives particles and vapors that are likely to be present on the exterior of a vehicle loaded with explosives. Our concept is to design an analysis fixture for a robotic arm that we can position close to various surfaces of a vehicle suspected of carrying a bomb. This fixture would include a device to collect particles from the surface under inspection, a sample preconcentrator, and an optical assembly to generate a laser induced plasma. The LIBS sensor would include a small laser, a spectrometer (located at the base), and a fiber optic bundle to transmit the laser induced atomic and diatomic emission produced from the sample to the spectrometer.

LIBS is well suited for our intended application. The hardware is robust (it does not require sensitive alignment); no sample preparation is required; and the analysis is rapid, straightforward, and has high sensitivity. Selectivity, however, is a potential problem since LIBS has been developed primarily for chemical elemental analysis; and simply determining the elemental composition of a sample alone is not sufficient to determine if it is an explosive. Therefore, improving the selectivity of LIBS as applied to explosives detection is, and continues to be, a major objective of our overall effort.

## Accomplishments

Our milestones for FY 2005 were to examine sensitivity, specificity, and possible interferents as related to LIBS analysis.

To improve the LIBS shot-to-shot reproducibility and to improve the signal-to-noise ratio, we made several modifications to the existing LIBS instrument. We motorized the sample platform and enclosed it for safety reasons and to provide purging environments.




*Laser sample shot.*

We evaluated two different spectrometers for use with the LIBS instrument. The first spectrometer was an échelle system, which provided sensitivity over a continuous range from 200 to 850 nm at moderate spectral resolution. The second spectrometer, a switchable grating system from Acton Research Corporation, was sensitive from 200 nm through 900 nm, but it could only capture a portion of the 200 to 900 nm spectral region at a time. Nevertheless, we decided to use the Acton spectrometer because of its enhanced sensitivity even though it restricted the width of the spectral region we could capture.

For LIBS selectivity, we believe we can use the ratio of the oxygen-to-nitrogen (O-to-N) line emission intensities to identify the presence of an explosive. However, it is possible that the presence of oxygen and nitrogen would interfere with accurate determination of this ratio if the sample is in air. To address this concern, we performed experiments using different purge gases and observed changes in the nitrogen and oxygen emission lines. We found changes in the relative intensities of the nitrogen and oxygen emission lines, as expected based on the purge gases (nitrogen, air, helium, and argon). Fortunately, the changes were not so great as to invalidate the O-to-N ratio as an indication of the presence of explosives. Nevertheless, we will conduct additional experiments to better understand the influence of air on the LIBS measurement of explosives.

Additional possible interferents include dust and/or road grime typically found on vehicles. Our initial road grime and dirt measurements indicate the presence of alkali and alkaline earth salts that produce a strong metal-atom emission, but the emission appears in a different wavelength region and will not likely interfere with the O-to-N ratio analysis. Preliminary analysis indicates that we are able to distinguish TNT from blanks and road grime.

The sensitivity and specificity of LIBS analysis are ultimately tied to the intensity of the specific atomic/molecular emission lines and their ratios utilized in the analysis. Therefore, we began experiments to determine the most suitable emission lines for analysis. Currently, the 390 nm and 765 nm spectroscopic regions seem promising due to the strong atomic and molecular emission lines that appear in these regions. We also examined the effect of different laser pulse-to-detection time delays and various temporal sampling windows (exposure times) for the carbon emission line intensity since it may be very useful in the specificity analysis. Additionally, our preliminary analysis of cyclotrimethylenetrinitramine (RDX), 2,4,6-trinitrotoluene (TNT), cyclotetramethylene-tetranitramine (HMX), pentaerythritol tetranitrate (PETN), and ammonium nitrate soaked with fuel oil (ANFO) show some differences in the 390 nm region for cyanide radical formation and subsequent emission for the different energetic materials.

Our decision to use the more sensitive spectrometer (with its restricted spectral coverage) did not permit the collection of all the elemental composition information that is potentially possible from a single laser pulse. Due to shot-to-shot variability of the plasma emission, the data from different laser shots could not be meaningfully compared. This situation limited us to the analysis of lines that occur within a single spectral window, when, in fact, some of the more meaningful data involve two or three spectral windows. Unfortunately, this instrument limitation kept us from performing more than preliminary analysis on milestones pertaining to the sensitivity and specificity of the LIBS technique. However, we ordered a new instrument with enhanced sensitivity that can acquire the plasma emission over the entire 200 to 900 nm range on every laser shot. This instrument will provide us with significantly enhanced analytical capability.

# Assessment of the Next Generation of Preboot Firmware

*79853*

*T. Q. Thai, M. A. Leger, A. Phan, N. S. Rattan, H. Brower, J. Z. Liang*

This is a classified report.

# Discrete Field-Portable Identity Microarrays

*79854*

*S. M. Brozik, P. L. Dolan, M. Manginell, J. H. Flemming*

## Abstract

We are working to develop a sensing system for human identification using deoxyribonucleic acid (DNA) fingerprinting technologies with microneedle array technology. Successful completion of this project will put Sandia at the forefront for providing key sensor technologies for discrete, field-portable human identification. At the heart of our approach is the use of the DNA fingerprinting technology, single nucleotide polymorphisms (SNPs), which are DNA sequences inherently unique to an individual.

In FY 2005, we designed and fabricated microneedle arrays in Foturan® and silicon to collect human skin cells. We tested Foturan needles plated in nickel, gold, and rhodium under shear forces up to 60 mN, and they remained intact after testing cell collection from pig skin. We used the Foturan needles/posts to collect cells from porcine skin. We lysed cells attached to the microneedles by applying a pulsed high potential or using impedance spectroscopy at low frequencies (1000–10 Hz) and at applied potential between 0.1 and 0.5 V. We also investigated a chemical means of cellular lysis using sodium dodecyl sulfate, which proved to be more practical. Using both optical and electrochemical methods, we demonstrated detection of hybridized DNA to its peptide nucleic acid (PNA) target. For optical detection, we designed the DNA molecule, complementary to the PNA, with an Alexa-Fluor 488 dye conjugated to its 5' end. The dye is visible at the sensor surface upon hybridization. For the electrochemical detection scheme, we developed a diazonium chemistry to indirectly attach the PNAs to the electrode surface. We investigated a new approach for assembling the diazonium layer using cyclic voltammetry. This work led to submission of a technical advance. We also optically demonstrated hybridization using a ribozyme sensor for DNA detection. The detection limit of this method was between 1.25 femtomoles and 500.5 attomoles of target DNA (~3–7.5 x $10^8$ molecules). Early detection within 5 minutes was observable, with the strongest signal occurring within 10–20 minutes.

## Accomplishments

(1) Demonstrate PNA/DNA hybridization within 40 minutes: One of the primary goals of the research team was to demonstrate that we could carry out PNA/DNA hybridization in less than 40 minutes. In order to demonstrate the hybridization of PNA and DNA oligomers, we needed to develop the chemistry to immobilize the PNAs on the electrode surface. We bonded a tightly packed monolayer of n-(4-aminophenyl)maleimide to a platinum surface by converting it to a diazonium salt. Through cyclic voltammetry, we attached the diazonium to the electrode one monolayer at a time; the more cycles, the thicker the film. We designed a custom PNA oligomer with a flexible chain at the 5' end terminating with a cysteine. The cysteine has a free thiol group that is available to react with the maleimide monolayer, thus attaching our PNAs indirectly to the surface.

(2) Investigate optical and electrical detection schemes for the hybridization reaction: Since the PNAs are quite expensive, we initially used aptamers and their target protein to demonstrate binding. We detected the presence of the protein, thrombin, bound to thrombin aptamers in less than 10 minutes electrochemically. To demonstrate detection of nucleic acids, we designed deoxyribozymes to bind a specific target sequence. We optically observed hybridization in 5 minutes, with a stronger signal after 10–20 minutes. The detection limit of this method was between 1.25 femtomoles and 500.5 attomoles of target DNA (~3–7.5 x $10^8$ molecules).

(3) Fabricate 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 Foturan needles sheared with 2 mN applied force, while the plated devices sheared at forces of 50–60 mN. We tested the 300 micron devices on pig skin. We collected dead cells in a scraping method. Even after we applied excessive force, the

device remained intact with no shearing of the individual needles.

(4) Demonstrate lysing of cells: We investigated two techniques to lyse cells attached to microposts fabricated from silicon and Foturan: (a) pulsed high potential cellular lysis in solution and (b) impedance spectroscopy. Using the first method, we successfully lysed epithelial cells; however, due to the large potential used, a significant amount of hydrolysis occurred, destroying the buffer that housed the cells.

Utilizing impedance spectroscopy, we applied a large alternating current field, up to the megahertz regime, between the microposts causing the charged particles inside the cell to migrate towards their respective ends of the cell, forming a dipole, and eventually ripping the cells apart. Although preliminary results were promising, we determined that running a design of experiments on potential and frequency would take a considerable amount of time and resources. Therefore, in the end we decided it was best to pursue a chemical means of cellular lysis using sodium dodecyl sulfate.

(5) Determine the DNA content of cells collected from the microposts: In order to determine the number of cells we would need to capture for an adequate signal, we had to determine the total amount of DNA in each cell. We used normal human epidermal keratinocyte cells as our model culture system for these experiments, as they are of the same cell type as would be collected using the microneedles. We used the DNeasy® Tissue Kit to extract the total DNA from the cultured cells. The protocol involves harvesting the cells, chemically lysing them, followed by DNA extraction. We then analyzed the DNA using a spectrophotometer. The amount of DNA measured was approximately three micrograms of DNA for every $1x10^6$ cells.

# Profile Based Network Anomaly Detection

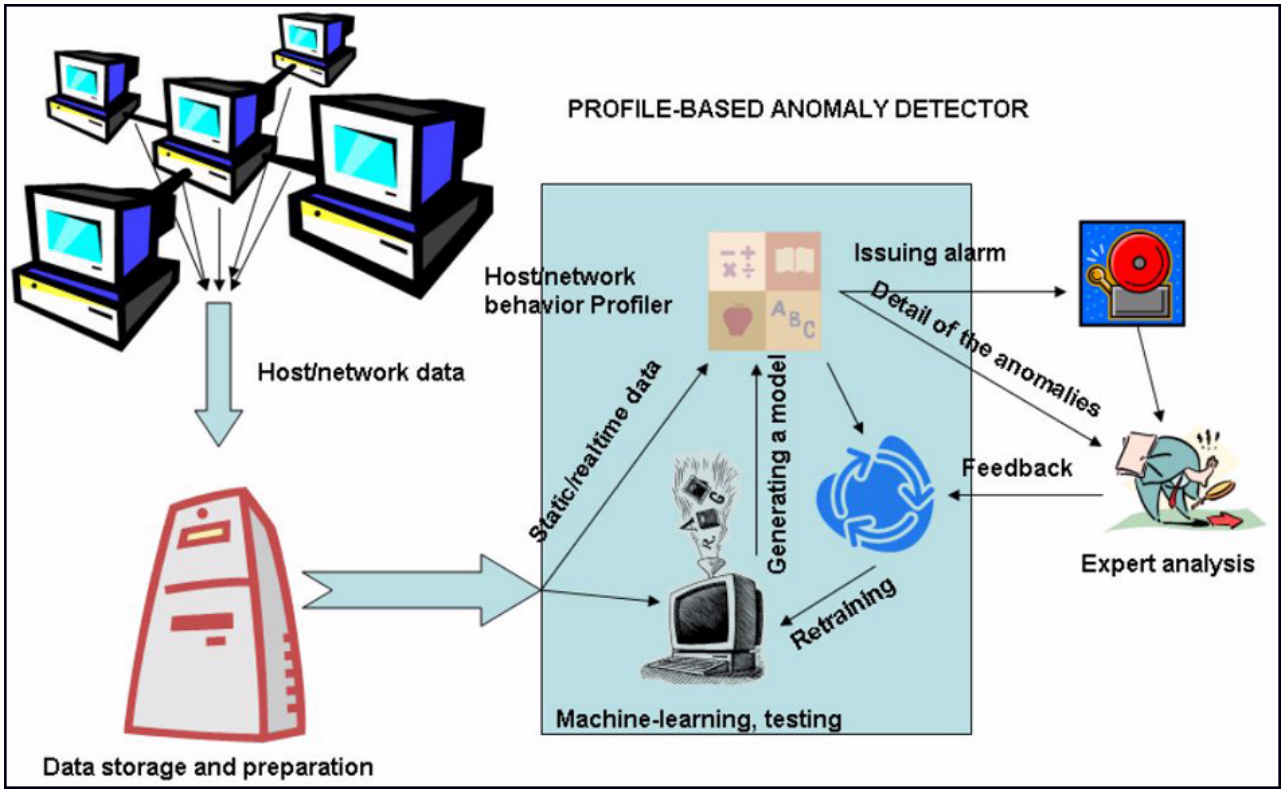
*79855*

*N. A. Durgin, G. N. Conrad, B. J. Tejani, P. Zhang*


## Abstract

As information systems become increasingly complex and pervasive, they become inextricably intertwined with the critical infrastructures of national, public, and private organizations. The problem of recognizing and evaluating threats against these complex, heterogeneous networks of cyber and physical components is a difficult one, yet a solution is vital to ensuring security. The purpose of this project is to research profile based anomaly detection techniques that can be used to address this problem. We focus primarily on the area of network anomaly detection, but will also investigate extending our techniques to other problem domains. We will investigate using several data analysis techniques to create profiles of network hosts to enable anomaly detection. The "profiles" will reduce multidimensional vectors representing "normal behavior" into fewer dimensions, allowing pattern and cluster discovery. We will compare new events against the profiles, producing a quantitative measure of how "anomalous" the event is.

Most network intrusion detection systems (IDSs) detect malicious behavior by searching for known patterns in the network traffic. This approach suffers from several weaknesses, including a lack of generalizability, an inability to detect stealthy or novel attacks, and lack of flexibility regarding alarm thresholds. Our research will focus on enhancing current IDS capabilities by addressing some of these shortcomings. We will identify and evaluate promising techniques for data mining and machine-learning. We will "train" the algorithms by providing them with a series of data points from "normal" network traffic. A successful algorithm, trained automatically and efficiently, will have a low error rate (low false alarm and miss rates) and will be able to identify anomalies in "pseudo real time" (i.e.,


*Anomaly detection architecture.*

while the intrusion is still in progress rather than after the fact).

We will design a lightweight, scalable, and distributable architecture we can generalize to other problem domains, enabling data fusion and knowledge discovery.

## Accomplishments

(1) We examined multiple data mining and machine-learning algorithms and evaluated their suitability for this problem. We selected self-organizing maps (SOMs) and adaptive resonance theory (ART) for detailed evaluation.

(2) We performed preliminary testing to provide a proof-of-concept that we can use host profiles created from high level TCP/IP (transmission control protocol/internet protocol) session data to distinguish between different hosts, and thus can also use them as components of an anomaly detection system.

(3) We are working with SOM and ART toolboxes and test data sets. We developed specific test vectors and methods for extracting datasets for model training, testing, and validation.

(4) We developed prototype programs, including graphical user interface components, to demonstrate how we can deploy the profile based anomaly detection and apply it to a network for monitoring in an intrusion detection system.

(5) We investigated issues concerning integrating the results of this research with other information operations (IO) research projects and technologies currently under development at Sandia.

(6) We built an anomaly detection simulator. This simulator can display the behavior features of a host and generate data files that represent the altered host behaviors at various time intervals. We integrated the anomaly detection protocol into this simulator. Thus, this simulator serves as a tool demonstrating the mechanisms and application of anomaly detection in network security.

## Refereed

N. Durgin, Y. Mai, and J. Van Randwyk, “NetState: A Network Version Tracking System,” presented and in *Proceedings of the Freenix/Open Source Track 2005 USENIX Annual Technical Conference*, Anaheim, CA, April 2005, vol. 1, pp. 119–127.

# Integrated Optical MEMS Using Through-Wafer Vias & Bump Bonding

*79856*

*F. B. McCormick, S. K. Frederick, A. H. Hsia, R. A. Haltli, O. B. Spahn, G. R. Bogart, W. D. Cowan, J. G. Fleming, B. Jokiel Jr., R. J. Shul, D. J. Dagel, T. Lemp*

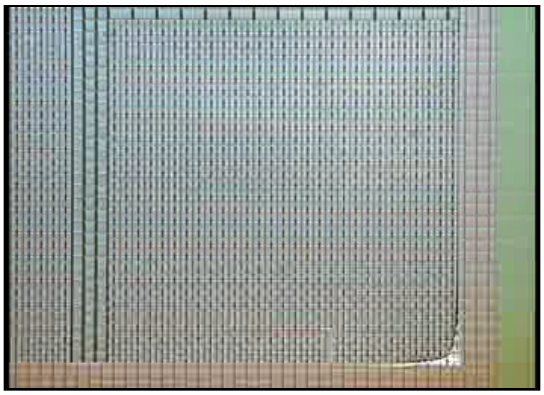
## Abstract

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 build large micromirror arrays that will improve optical target identification capability by a factor of three. 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 polysilicon/metal interconnects and matching metal bumps. It 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. In contrast, our proposal bypasses these limitations and compromises by realizing through-wafer vias that are compatible with Sandia Ultraplanar Multilevel MEMS Technology 5 (SUMMiT-V™). To fully develop and demonstrate this integration process, we will leverage the mature SUMMiT-V MEMS process to fabricate and test a 4096 OMEMS mirror array and bond it to a suitable addressing/control integrated circuit. Our progress in demonstrating a stable through-wafer via process and bump bonding attachment success provides us confidence in achieving this goal.

## Accomplishments

We made substantial progress within the five key areas of this project: (1) through-wafer vias, (2) pixel control and drive electronics, (3) MEMS chip-to-electronics chip attachment, (4) optical MEMS array test and characterization, and (5) overall microsystem integration. We developed a novel high density/voltage (> 100 V) through-wafer via process flow with high yield and submitted a technical advance. This new process requires no wafer thinning and is fully compatible with subsequent SUMMiT-V fabrication processes for MEMS or other sensor structures. This high density process allows completion of the vias with material sets such as polysilicon, aluminum, tungsten, gold, copper, and platinum, as well as arbitrary backside bond-pad definition using a new dry film photoresist approach. We demonstrated a via test approach using visual inspection of MEMS mirrors to validate via continuity and yield. We performed pixel driver circuit design analysis to compare footprints, speed, power dissipation, and complexity. We conducted a search for a multiproject foundry offering high voltage (> 30 V) circuit processes and selected PolarFab and AMI (primary and backup) to support our initial test chip fabrication runs. We identified a key incompatibility between our circuit design tools and the foundries' high voltage design rule software and developed a path to address this hurdle. We investigated several solutions for the bump attachment process, and we selected a process using low temperature conductive epoxy on gold bumps over a higher temperature solder reflow approach.



*Video clip showing demonstration of 32x32 MEMS mirror array activation using through-wafer vias.*

We created a test vehicle consisting of three different via test structures. We identified external vendors as well as internal Sandia options for high density bump creation, and we demonstrated epoxy coated bumped substrates. To protect the driver circuits from

the MEMS release process, we characterized four different acid resistant underfill epoxies; we found Epotek 377 to perform very well against the release chemistry. We designed and built an adaptive optics testbed that allows demonstration of the wavefront correction function of MEMS mirror arrays and includes a software interface that allows addressing of the control board that carries the packaged MEMS chip. We conducted a risk assessment to investigate bump bonding metallurgy issues, processing temperatures on mirror figures, bonding alignment and assembly issues, large array test processes and data collection, etc. We developed mitigation plans to address these concerns to avoid problems and delays at the microsystem integration level. The demonstration of high yield vias through unthinned wafers, together with the progress in bump bonding and electronics design, leaves us well positioned to achieve our overall project goals.

# Defense Against Future Cyber Agents

*79857*

*S. Y. Goldsmith, S. V. Spires, D. A. Rieb, J. A. Hutchins, R. C. Parks, R. E. Carlson*

## Abstract

The maturation of intelligent agent technology (IAT) has raised concerns in various sectors of the US government about its use as an advanced cyber weapon. These concerns are centered on the possibility of misusing software agents to attack computers and computer networks on a larger scale and with more devastating effect than is possible with existing cyber attack tools. We determined some general issues and performed initial research regarding desired criteria for defensive agent systems, models of cyber agent collectives, and detecting coordinated attacks from organized collectives of cyber agents. The detection technique investigated is based on a support vector machine learning algorithm applied to a packet based simulated intrusion detection system.

## Accomplishments

(1) Needs Assessment

We conducted a needs assessment that maps current issues and problems in the domain of cyber defense to agent systems and technology. The analysis resulted in a concept of operations, and a suite of potential use cases, and identified a demonstration prototype that captures the distinctive features of agent technology to the problem. We developed a demonstration scenario to exhibit the prototype system in operation and initiated development of the prototype.

(2) Agent Modeling

We developed stochastic models representing software agents situated on a network in an agent vs. agent conflict. We used the models to gain insight into the management of uncertainty in a defensive scenario involving several thousand agents. We used the models in an interacting particle simulator to gain an understanding of population level dynamics of two teams of competing agents. The results of the simulation provided information on sensitive parameters and on the computational complexity and scalability of the approach.

(3) Coordinated Attack Detection

Through a collaborative student project with the University of Wisconsin and the University of Minnesota, we investigated an initial approach to detecting coordinated cyber attacks based on a support vector machine learning algorithm.

# Infrastructural Development for Wireless Devices

*79858*

*M. H. Johnson, D. J. Wiener, J. D. Tang, K. B. Kominek, L. G. Martinez Jr., J. T. Gardner, N. Dautenhahn*

This is a classified report.

# Stressed Glass Technology for Actuators and Removable Barrier Applications

*79859*

*S. J. Glass, A. S. Tappan, K. J. Schwing*

## Abstract

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 to mitigate interference with the function or motion of other parts. Brittle materials like glass, especially glass that is strong or pre-stressed, are ideal candidates for these applications.

Glass can be produced in different sizes and shapes. We can manipulate the strength and pre-stress, which control the fragment size by either tempering or ion exchange processes. We can harness the energy stored in the residual stress profile to drive fragmentation of the component once it fractures. We can also store energy in the glass component, in the form of stored elastic strain energy, by mechanically loading it. Due to the high strength of stressed glass, we can store and then release a significant amount of energy, either to perform useful work or to initiate another action.

Our objectives are to advance stressed glass processing, to develop prototype devices, and to demonstrate their potential. Work this year included identifying and designing several prototype applications. Due to the need for more complex shapes, 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. We also directed efforts toward methods for weakening the glass in selected regions.

## Accomplishments

The first project milestone was to obtain more information about applications of interest. We conducted a literature review to identify previous research for several potential applications, but information was limited. The second milestone was to prioritize the applications based on potential customer needs, and to determine the feasibility of using stressed glass for each of the applications. The feasibility of making applications out of stressed glass relates to the form and sizes of glass that are available, processing (times and temperatures), and the performance criteria. Complex shapes require machining, bonding individual glass pieces, or molding or shaping the glass. Due to the residual stress profile in the glass produced by an ion exchange treatment that is a diffusion controlled process, more complex shapes also lead to different stress profiles in different parts of the glass. Adjusting the level of surface compression and internal tension, respectively, controls the glass strength and fragmentation properties. We can control the surface compression and tension by changing the ion exchange time and temperature and the glass thickness and composition. Most of the work for this project used a sodium alumino-silicate glass composition.

The performance criteria for the applications are: that the glass have a specific strength, that it break into fragments with a controlled size, and that the glass satisfies optical or electrical requirements. The applications selected include a device that acts as a stress/shock sensor with directional discrimination capabilities, and a glass plate with electrical lines. The shock sensor design requires bonding a glass rod with a reduced section to a flat plate of the glass. During shock loading, the reduced section of the glass rod experiences elevated stress and is expected to fracture at a specific G load. We evaluated glass bonding methods, including a new low temperature (room temperature to ~120 °C) technique developed by SCHOTT.

We investigated methods for releasing the energy that is stored in the tensile region of stressed glass. Our tests included mechanically inducing fracture with a hardened punch on a pendulum and using small amounts of explosive materials in contact with a glass disk. Our mechanical initiation of fracture included high speed photography characterization of what happens to the glass fragments after the crack propagates across the disk. Significantly more fragments are

launched from a stressed glass sample than an as-received sample. Our efforts to initiate fracture using explosive materials were unsuccessful; it appears that the peak energy is insufficient even to break an as-received glass in this configuration. We made new parts in which we drilled holes in the glass to contain the explosive and increase the amount of energy transmitted to the glass to break it.

One of the constraints on the use of stressed glass is that it is so strong after the ion exchange treatment that it is very difficult to initiate the fragmentation process. To overcome this problem, 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 successfully blocked the ion exchange process and changed the stress profile in the glass. We used finite element analysis to model how preventing ion exchange in localized regions changes the residual stress profile.

## Other Communications

M. Abrams, "Crack Propagation and Fracture in Engineered Stress Profile Glass," Pennsylvania State University PhD Dissertation, University Park, PA, 2005.

J.E. Kooi and S.J. Glass, "Crack Branching Density in Ion-Exchanged Glass," presented at the 16th Annual Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2004.

S.J. Glass, "Glass Fracture: Can We Control It?" presented at New Mexico Tech Invited Seminar, Socorro, NM, November 2004.

S.J. Glass, "Glass Fracture: Can We Control It?" presented at an invited seminar at the University of New Mexico, February 2005.

S.J. Glass, "Glass Fracture: Can We Control It?" presented at the Architectural Surety Team Meeting, Chavez Grieves Engineering, May 2005.

S.J. Glass and R. Tandon, "Processing and Properties of Ion Exchanged Glasses," presented at the 16th Annual Rio Grande Symposium on Advanced Materials, Albuquerque, NM, October 2004.

J.L. Rygel, R. Tandon, M. Braginsky, and S.J. Glass, "Simulation of Fracture Patterns Obtained During Fragmentation," presented at the 107th ACerS Annual Meeting, Baltimore, MD, April 2005.

R. Tandon, "Fragmentation in Brittle Materials: Existence of an Energy-Based Limit," presented at the Fracture and Flow of Glass Conference, College Park, PA, 2005.

R. Tandon and S.J. Glass, "Controlling the Fragmentation Behavior of Stressed Glass," presented and in *Proceedings of the 8th International Symposium on Fracture Mechanics of Ceramics, Active Materials, Nanoscale Materials, Composites, Glass, and Fundamentals*, Houston, TX, February 2003, vol. 14, pp. 77–91.

J. Rygel, R. Tandon, S.J. Glass, and M. Braginsky, "Simulation of Fracture Patterns Observed During Fragmentation," presented at the Fracture and Flow of Glass Conference, University Park, PA, October 2005.

# The Surety of Cyber Tools

*79860*

*M. D. Torgerson, K. O. Ramotowski, D. P. Duggan, W. D. Neumann, L. G. Pierson, P. L. Campbell, R. C. Schroeppel, H. E. Link, W. E. Anderson*

## Abstract

In today's world, the continual use of cyber equipment is a way of life. An integral part of the cyber world is the collection of software tools that make the cyber world viable. Unfortunately, there are certain adversaries that make protecting one's cyber assets difficult. The premise of this work is that, as a matter of course, one will develop cyber tools they can use in their own networks. Those tools are valuable assets and should have their own protections.

The cyber tool protections may take on many different guises, all of which must be carefully designed and controlled. This project is an effort to understand the protection of cyber tools, to develop methods for that protection, and to prototype those methods. We call these protections and methods use-control of cyber tools.

## Accomplishments

To better understand issues related to the use-control of cyber tools, our first milestone was to define use-control areas and examine lifecycle and threat model issues. We accomplished this by examining various lifecycle models, differentiating use-control measures needed during different lifecycle stages, and examining the open literature for known use-control techniques. We documented our results, including a taxonomy of use-control techniques classified by the service provided. Given a cyber tool, we can create a matrix listing lifecycle phase and necessary security services. We can consult the taxonomy of use-control methods to see if suitable techniques exist to achieve the desired service appropriate for the lifecycle phases. By completing the matrix (or not), we can determine if security is adequate for the cyber tool.

A second goal was to conduct meaningful research into areas adding value to existing use-control measures or creating new techniques not found in the taxonomy. Our focus areas were secure obfuscation, multiperson control, an authenticated access control mechanism, and faithful execution techniques using an obfuscated decryptor. We documented an obfuscation method and developed an obfuscated password controlled access mechanism.

A third goal was to assess the appropriateness and efficacy of the use-control measures in real cyber tools. We created a "red teaming" tool that comes in two parts. The first part, Program A, is an intelligence gathering tool that identifies a target machine address with a specific property. Program B uses this information to execute a traffic magnification program that causes denial of service. We demonstrated the ability (using trusted hardware) to maintain integrity and execution control of Program A. We demonstrated a secure, remote, obfuscated two person execution control of Program B. Further, we maintain confidentiality of both programs before execution.

# Terahertz Quantum Cascade Lasers for Standoff Molecule Detection

*79861*

*M. C. Wanke, M. A. Mangan, C. T. Fuller, J. L. Reno, W. W. Chow, J. D. Blaich, E. W. Young, S. Samora*

## Abstract

Sources of coherent THz frequency (i.e., 0.1 to 10 THz) electromagnetic radiation with requisite power levels, frequency agility, compactness, and reliability represent the single greatest obstacle in establishing a THz technology base. To be truly useful, a coherent THz source should be as compact, reliable, and frequency versatile as familiar solid state radio frequency/microwave and infrared/visible source technologies, and be able to deliver average power at least in the 10 to 100 mW range. Current generally available THz source technologies can usually deliver only some of the features called for in conceivable THz applications, and thus severe and highly constraining compromises must be made. Recent advances in semiconductor based quantum cascade lasers (QCLs) offer huge improvements towards the ultimate THz source goals.

We are developing narrow linewidth THz quantum cascade lasers to enable optical chem/bio detection capabilities for homeland defense and nonproliferation. Molecular spectral modes in the THz 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. We will develop and use theoretical tools to guide the investigation of nonlinear optics, employing infrared QCLs and the exploration of quantum coherence to improve QCL performance, especially towards high temperature operation. In conjunction, we will create novel direct THz QCL designs to increase output power.

## Accomplishments

- *Theory and Modeling:* In order to get first insights into the influence of bandstructure on the output of QCL lasers, we developed a free-carrier model for the QCL gain region. Based on experience with conventional interband lasers, Maxwell equations for the laser field and semiconductor Bloch equations for the gain material are solved simultaneously in the time domain. The equations of motion are k-resolved due to different effective masses or a nonparabolic curvature (source for inhomogeneous broadening) with respect to a possible k-dependent transition energy between the laser subbands. Additionally, this k-resolved treatment enables us to investigate nonequilibrium effects. This treatment goes well beyond what other researchers in the field have explored, and addresses critical issues for device understanding and simulations.
- *THz Laser Design:* We enhanced our Schroedinger-Poisson solver to handle the more demanding requirements of THz lasers over mid-infrared lasers, most notably adding in periodic boundary conditions and non-Fermi level distributions to the Poisson solver. In addition, we developed a novel emitter design that may enable room temperature THz operation, and submitted a technical advance.
- *Development and Testing:* We finished the development of a reliable robust fabrication process for THz quantum cascade lasers, and demonstrated reproducible THz laser performance. This completed all the necessary requirements for THz laser development (modeling tools, growth, fabrication, and testing capabilities) and now allows us to focus more on laser development.

## Refereed

I. Waldmueller, W.W. Chow, M.C. Wanke, and E.W. Young, “Semiclassical Many-Body Theory of Quantum-Cascade Lasers,” presented at Intersubband Transitions in Quantum Wells, North Falmouth, MA, September 2005.

# Advanced Technologies for National Security Applications

*79862*

*A. C. Hall, R. S. Bennett, M. C. Jackson, J. C. Siemers, G. R. Anderson, M. C. Kidd*

## Abstract

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 will combine and leverage four diverse areas of Sandia expertise and technology to create a formidable new protective system. Development of this concept can only be accomplished at a national security laboratory like Sandia. The key technologies involved are: (1) microsystems, (2) materials science, (3) software, and (4) vulnerability analysis. Individually, these technologies have been applied to protective solutions, but they have never been integrated in the synergistic manner investigated in this project.

Technical challenges to implement this concept 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.

## Accomplishments

We completed successful proof-of-concept experiments and further development work is under way. We held brainstorming sessions that resulted in a number of new ideas for protective technology solutions.

# Forensic Tool Development for SCADA Systems

*79863*

*M. J. Berg, A. Berry, B. T. Richardson, J. Trent, K. L. Loesch, M. J. Baca, E. J. Hokanson, K. S. Nauer, D. P. Duggan, R. E. Carlson, J. J. Torres, P. G. Cordeiro, J. E. Stamp, M. A. Rumsey, T. J. Draelos*

## Abstract

The US critical infrastructure relies on supervisory control and data acquisition (SCADA) systems for control, data collection, emergency response, and safety response. These systems are now more vulnerable to attacks due in part to their integration with corporate business networks and their migration to, and close integration with, general purpose information technology (IT) networks and systems.

However, the state of the art in SCADA systems is often lacking security mechanisms. SCADA specific forensic capabilities are practically nonexistent. As the risks to these systems from both inside and outside attack increase, the need to determine what, when, and how an attack occurred, and who was involved, becomes critical.

Forensic systems capable of dealing with SCADA specific system properties need to be developed to address this issue, to better understand the unique security risks faced by SCADA systems, and to understand how to mitigate those risks. This project will develop tools and techniques for performing forensic analysis on SCADA systems so that we can study intrusions into these systems and prevent future intrusions.

## Accomplishments

We surveyed the capabilities of a number of existing digital forensics tools. We selected the EnCase Enterprise Edition package from Guidance Software due to its extensibility and its ability to capture forensic images of operational systems, and current market share within corporations and law enforcement agencies.

We identified the special features requiring the extension of forensic tools within SCADA systems as well as the special SCADA software running on the operator consoles and the embedded systems such as programmable logic controllers (PLCs) and remote telemetry units (RTUs) that monitor and control physical processes.

We wrote a series of EnScript programs to allow EnCase to parse and analyze the configuration, log, and data files for one SCADA software suite. The techniques and programs developed this year will serve as the foundation for creating forensic analysis programs for additional SCADA software suites.

We created a database of checksums and file information for several popular SCADA software suites. We can use this information to quickly identify modifications to the original software during an investigation. We developed the ability to generate and maintain this information in order to facilitate better security and audit capabilities within operational SCADA systems.

We implemented a prototype tool to capture forensic images of the configuration and firmware for one series of PLCs. This tool allows us to compare the PLC's configuration and firmware against a baseline image to identify alterations made to the device. We will apply the experience gained in the development of this tool to capturing forensic images from other PLCs and RTUs.

# Verification Through Process Monitoring

*79864*

*P. E. Rexroth, B. B. Cipiti*

## Abstract

Current reprocessing plants have a stringent system of process controls and safeguards in place to control nuclear materials. These controls keep track of nuclear material for safe operations and for prevention of material loss or diversion. However, the traditional data acquisition process for material accountability is often not optimized for cost or effectiveness for either the operator or inspector. We developed a process and instrumentation model of a generalized PUREX reprocessing facility to examine fault detection and to optimize sampling strategies for both operator and inspector owned monitoring equipment. The unique aspect of our work is that the material quantity at any place and time is a function of the material balance histories throughout the system. We developed measurement error and detection probability models and will use them to evaluate instrumentation deployment scenarios.

## Accomplishments

(1) We developed a process and instrumentation model of a generalized PUREX reprocessing facility. The reference process for our work is used at the Barnwell reprocessing facility. Our model includes some 20 major unit operations, resulting in more than 200 differential mass balance equations to be solved simultaneously. We based operating parameters, such as separation efficiencies and tank discharge rates, on material balance information provided in the literature for processing 1500 metric tons of heavy metal per year. We prepared a slight modification of the model to also model the thermal oxide reprocessing plant (THORP) in the United Kingdom.

(2) We developed measurement error models that treat random and systematic errors in either an additive or a multiplicative manner.

(3) We developed a detection probability model that is based upon the two sided Page's test. We are constructing an alternative formulation based upon the Kalman filter method. We are benchmarking the models against reported results from near real time accounting experiments performed at THORP.

## Refereed

P.E. Rexroth, D.S. Blair, B.B. Cipiti, N.L. Ricker, and B.A. Purvis, "Verification Through Process Monitoring," to be published in *Proceedings of the INMM 46th Annual Meeting*, Phoenix, AZ, July 2005.

# Monolithically Integrated Microcoolers for Sophisticated Sensor Systems

*79865*

*J. K. Kim, J. L. Rienstra, M. S. Carroll, S. D. Mukherjee, G. A. Vawter, A. J. Routson*

## Abstract

The issue of cooling focal plane arrays (FPAs) constrained by the stringent size and power budget of a satellite must be addressed to continue the push toward higher sensitivity and resolution infrared (IR) imaging. We expect thermionic coolers to be as much as five times more efficient than currently used coolers. Thus, they are suitable for addressing this issue, especially when employed to selectively cool the FPA in an integration scheme involving the FPA and the supporting electronics/photonics elements. In addition, semiconductor thermionic coolers are robust, compact, and reliable. However, thermionic coolers are generally less efficient at cryogenic temperatures, and they are difficult to scale to large sizes without sacrificing performance. This project aims to address these issues and to develop thermionic coolers that are best suited for cooling large area FPAs in a scheme conducive to further integration with electronic and/or photonic circuits.

We quantified the need for a substantial reduction in the power required to cool mid-IR sensors that will enable the next generation of space based imaging systems. We identified and evaluated the most likely candidate technologies to deliver such a breakthrough. We quantified the enhanced cooling efficiency benefits of thin film, solid state based thermionic coolers. We also identified and investigated a novel mid-IR detector concept that can dramatically increase the mid-IR sensor operating temperature, thereby solving the cooling power problem. We laid the groundwork for a full investigation of this type-II superlattice detector technology.

## Accomplishments

We investigated the problem of IR FPA cooling constrained by the stringent size and power budget of a satellite to generate solutions that facilitate the continued upward performance and functionality scaling of satellite based IR imaging systems. After thorough comparisons with competing technologies, we identified and analyzed two novel solutions that promise to cut the cryocooler power requirements in half by enabling the imaging system to operate at a much higher temperature. Incorporating thin film thermionic coolers with FPAs and/or developing long wave infrared detectors that perform well at 200 K can permit ~50% cooling power reduction and thereby allow larger/additional sensors for enhanced functionality and performance, or allow cheaper and lighter sensor systems. We studied device performance optimization concepts and material optimization parameters in detail and drafted development paths for the two technologies. We also identified limits and risks of the technologies, as well as the corresponding mitigation plans.

# Tunable Dielectric Films for Frequency Agile RF and Microwave Integrated Circuits

*79866*
*C. D. Nordquist, J. Sigman, G. M. Kraus, P. G. Clem*

## Abstract

Future radar and communication systems, especially those using antenna arrays, will require frequency agile filters, tuning networks, and low loss phase shifters. Previously, these functions were achieved using bulky and heavy ferrite phase shifters or solid state microwave monolithic integrated circuit (MMIC) devices, resulting in high loss, narrow bandwidth, and poor linearity. While radio frequency (RF) microelectromechanical system (MEMS) technology has potential for low loss and high linearity, it suffers from limited continuous tuning range, low switching speeds, and poor reliability. Tunable dielectrics, such as barium strontium titanate (BST) based ferroelectric materials, allow tuning and reliability in conjunction with low power consumption and high switching speeds.

Electromagnetic simulations and measured results indicate that tunable filters and phase shifters are possible using these materials. Capacitance tuning ranges as high as 140% with acceptable Qs have been measured for simple planar interdigital test structures, comparing favorably to the 30% tuning range typically found in analog MEMS devices. Also, the material has a characteristic switching time on the order of nanoseconds, compared to microseconds for MEMS, which should enable fast reconfigurability and the rapid beam steering necessary for array antenna applications.

In this project, we are integrating the tunable dielectric films into Sandia's microwave integrated circuit process, and are evaluating the performance of RF circuit components realized in the integrated technology. We are examining the trade-offs between permittivity, loss, and tuning range for a wide range of tunable dielectric materials and device structures. We will design and fabricate circuits such as phase shifters, tunable filters, and tunable antennas capitalizing on those properties. At the end of this project, we expect to have a tunable dielectric microwave integrated circuit technology offering fast tuning, low loss, and high reliability.

## Accomplishments

In FY 2005, we met all milestones except the integration of superconductor films. We delayed the integration of superconductor films in order to focus on the thermal stability of the tunable dielectric films. We evaluated the dielectric tuning, temperature stability, and RF performance of a range of BST films ranging from 0 to 100% barium content. Results indicate that mid-range barium contents provide the best compromise between tuning range and loss for capacitors fabricated using these films. Additionally, we studied the dependence of the film performance on deposition parameters in order to enable temperature insensitive circuit tuning. We substantially improved the temperature dependence of these films through the use of multilayer composite films and lower annealing temperatures. We fabricated, tested, and modeled evaluation capacitors using these films, providing an understanding of the impact of capacitor geometry on its high frequency properties and allowing the design of several demonstration circuits. We designed distributed transmission line and reflect line phase shifters, as well as tunable end-coupled filters, using the extracted capacitor models. We demonstrated the process steps needed to integrate the film with the microwave integrated circuit process and realized simple single layer capacitors, paving the way for first-pass success for the microwave integrated circuit demonstration.

## Other Communications

J. Sigman, J.T. Dawley, J.J. Richardson, R. Ong, and P.G. Clem, "Epitaxial Growth of (Ba, Sr)$TiO_3$ Films on Ni(200) Tape Substrates," presented at the American Ceramic Society Annual Meeting, Baltimore, MD, April 2005.

J. Sigman, P.G. Clem, J.J. Richardson, and C.D. Nordquist, "Improved Dielectric Response and Temperature Stability in (Ba,Sr)$TiO_3$ Thin Films for Frequency Agile Microwave Integrated Circuits," presented at the 6th Pacific Rim Conference, Kapalua, HI, September 2005.

P.G. Clem, J.T. Dawley, J.A. Sigman, R.J. Ong, J. Richardson, C.D. Nordquist, and D.R. Tallant, "Integrated $SrTiO_3$ and $(Ba,Sr)TiO_3$ Tunable Dielectrics for GHz Frequency Agile Applications," presented at the American Ceramics Society Electronics Division Meeting, Cocoa Beach, FL, January 2005.

P.G. Clem, J.A. Sigman, J.J. Richardson, C.D. Nordquist, and D.R. Tallant, "Strain and Microstructure Effects in Chemical Solution Deposited (CSD) $(Ba,Sr)TiO_3$ Thin Films," presented at the American Ceramic Society 6th Pacific Rim Conference on Glass and Ceramics, Maui, HI, September 2005.

# Micropolarizing Device for Long Wavelength Infrared Polarization Imaging

*79867*

*S. A. Kemme, R. Boye, A. A. Cruz-Cabrera, T. Carter, J. L. Smith, J. R. Wendt, G. A. Vawter, C. Alford, G. R. Hadley*

## Abstract

The goal of this project is to fabricate the four-state pixilated 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 the 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 propose to fabricate an array of subwavelength components for MWIR polarization imaging applications. Each pixel unit of the array consists of four elements. Three of these elements are micropolarizers with three different polarizing axis orientations, and the fourth element is a micro birefringent waveplate on the top of another micropolarizer. The linear micropolarizers will be fabricated by patterning nanoscale metallic grids on a transparent substrate. The micro birefringent waveplate will be fabricated 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. The birefringent waveplate is a deeply etched dielectric one-dimensional subwavelength grating; therefore, two orthogonally polarized waves have different phase delays. Finally, we will investigate the near field and diffractive effects of the subwavelength element apertures upon detection.

## Accomplishments

We finalized the designs of the polarizer and the birefringent plate. We fabricated the polarizers on fused silica, and we began modeling near field effects. We moved the wavelength regime from the LWIR to the MWIR to leverage existing laboratory equipment. The dimensional features are more difficult to fabricate in the MWIR, given their smaller size due to the shorter wavelength, but still within our capabilities; and the results will also be valid for LWIR applications.

We designed wire grid micropolarizers for MWIR that will use lithium fluoride substrates. We designed and fabricated devices that have wire grids in gold of 0.2 micron width repeated every 0.4 micron, and a thickness of 0.15 micron plus an underlayer of titanium with the same pitch and period but with a thickness of 0.01 micron. To increase our confidence while waiting for the delivery of lithium fluoride (LiF) substrates, we fabricated and tested devices that work in the 2 to 3.5 micron range using fused silica as the substrate with the same design intended for lithium fluoride. We tested the devices and measured extinction ratios larger than 100:1 and transmission efficiencies for the transverse magnetic polarization larger than 80%. We made measurements on the polarizers at tilt angles between 0 and 30 degrees. In addition, we completed the design and the process development for quarter wave retarding devices. We first completed designs for the LWIR range and later added designs for the MWIR range.

As part of the analysis of diffraction effects on smaller aperture polarizers, we purchased and trained our team members on new software tools, Diffract and Sim3D-Max™, which are intended for this type of application. Diffract is a physical propagator intended for the analysis of coherent beams and their propagation though an optical setup. Sim3D-Max is a finite difference timed domain (FDTD) simulation tool intended for the near field analysis of the devices. These software tools are important, given that pixel sizes are in the order of three to seven waves and the proximity of one device to another one could be in the order of one to two waves. As expected, initial results from the FDTD indicate that diffraction effects from periodic small apertures will increase cross-talk as the distance between the polarizer and the active area of the focal plane array increases.

## Refereed

S.A. Kemme, D.W. Peters, J.R. Wendt, T.R. Carter and S. Samora, "Subwavelength Diffractive Optics for

Complete Micropolarizer Arrays in the Short-Wave and Mid-Wave IR," presented at the OSA Diffractive Optics and Micro Optics, Rochester, NY, October 2004.

S.A. Kemme, D.W. Peters, T.R. Carter, S. Samora, and J.J. Hudgens, "Subwavelength Structures for Micropolarizers and LO Applications," presented at the HAVE Conference, Lexington, MA, September 2004.

# Randomness Complexity and Cryptography

*81209*
*M. D. Torgerson*

## Abstract

As we rely more and more on digital communications, the privacy and security of our electronic data are essential. Sensitive military, financial, and personal information must be kept hidden from prying eyes; and we must ensure that honest parties can communicate securely and privately, even in the presence of malicious adversaries who will do anything to thwart their efforts. This goal of secure and private communication is the essence of cryptography. This research will concentrate on an interweaving set of research questions that all contribute to this goal by employing tools from the theory of derandomization.

This research will explore the applications of unconditional pseudorandom generators and answer the following questions: Can such tools be used in improving the security and/or efficiency of cryptographic protocols? Can such approaches be applied to practical cryptosystems, resulting in concrete performance gains? In light of the surprising efficacy of derandomization in hardness amplification, these are natural questions that will be answered in this research.

## Accomplishments

We explored the use of nondeterminism to amplify hardness and managed to amplify hardness within NP beyond the 1/2–1/n barrier. We showed that if NP contains a balanced function that is 1/poly(n)-hard for circuits of size s(n), then NP contains a function that (1/2–1/s'(n))-hard for circuits of size s'(n) for some function $s'(n) = s(\sqrt{n})\Omega(1)$. In particular, if $s(n) = n\omega(1)$, we amplify to hardness $1/2–1/n\omega(1)$ and if $s(n) = 2\Omega(n)$, we amplify to hardness $1/2–1/2\Omega(\sqrt{n})$.

In the field of computational complexity, this project has advanced the area of parallel complexity of performing computations in finite fields. Finite fields have a wide variety of applications in computer science, ranging from coding theory and communications to cryptography to complexity theory. We specifically focused finite fields of characteristic two; that is, finite fields GF(2n) having 2n elements. In particular, the question we addressed is: To what extent can basic field operations (e.g., multiplication, exponentiation) in these fields be computed by constant-depth circuits? We considered three natural classes of unbounded fan-in constant-depth circuits: circuits over the bases {AND, OR} (i.e., AC0), {AND, OR, Mod 2} (i.e., AC0[mod 2]), and {AND, OR, Majority} (i.e., TC0). We concentrated on the two following arithmetic problems: iterated multiplication and exponentiation in GF(2n).

We proved that DLOGTIME-uniform TC0 equals the class AE of functions computable by certain arithmetic expressions. This answered a question raised in the literature more than 10 years ago. We also showed how certain optimal constructions of k-wise independent and ε-biased generators are explicitly computable in DLOGTIME-uniform AC0[mod 2] and TC0. This addressed another question raised in the literature.

We introduce a new flavor of commitment schemes, which we call “mercurial commitments.” Informally, mercurial commitments are standard commitments that have been extended to allow for *soft* decommitment. Soft decommitments, on the one hand, are not binding but, on the other hand, cannot be in conflict with true decommitments. Specifically, we give efficient mercurial commitment schemes based on the discrete logarithm problem, claw-free trapdoor permutations, and the RSA problem. On the purely theoretical side, we show how to build interactive mercurial commitments from one-way functions, using the machinery of generic zero-knowledge proofs. Combining these constructions with our general framework for constructing zero-knowledge sets from mercurial commitments (plus collision-resistant hash functions), we produced zero-knowledge set constructions based on the hardness of factoring, the discrete logarithm assumption, the RSA problem, and general cryptographic assumptions. In fact, our discrete-logarithm zero-knowledge set construction is essentially based upon a published construction from the literature that inspired our definition of mercurial commitments. Our work can be viewed as a modular explanation, a simplification and a generalization of that work.

## Refereed

A. Healy, S. Vadhan, and E. Viola, "Using Nondeterminism to Amplify Hardness," submitted to *SIAM Journal on Computation, STOC2004 Special Issue*.

## Other Communications

A. Healy, "Constant-Depth Circuits for Arithmetic in Finite Fields of Characteristic Two," seminar presented at the University of Aarhaus, Aarhaus, Denmark, May 2005.

M. Chase, A. Healy, A. Lysyanskaya, T. Malkin, and L. Reyzin, "Mercurial Commitments with Applications to Zero-Knowledge Sets," presented and in *Proceedings of the 24$^{th}$ IACR Eurocrypt Conference (EUROCRYPT 2005)*, Aarhaus, Denmark, May 2005, CD-ROM.

# Electroforming of $Bi_{(1-x)}Sb_{(x)}$ Nanowires for High Efficiency Microthermoelectric Cooling Devices on a Chip

*84318*

*M. P. Siegal, W. G. Yelton, E. B. Webb III*

## Abstract

Active cooling of electronic systems for space based and terrestrial national security missions demands use of Stirling, reverse-Brayton, closed Joule-Thompson, pulse tube, and even more elaborate refrigeration cycles. Such cryocoolers are large, expensive high power systems often containing moving parts and are difficult to integrate with electronic systems. On-chip, solid state, active cooling can greatly enhance future system capabilities by reducing size, cost, and inefficiencies compared to existing solutions. We propose to develop a thermoelectric cooler capable of reaching 77 K by replacing bulk thermoelectric materials with arrays of BiSb nanowires. Furthermore, the Sandia developed technique we use produces oriented nanowires at room temperature that we can apply directly to a silicon substrate. Key obstacles include (1) optimizing the BiSb alloy composition for thermoelectric properties, (2) increasing wire aspect ratios to 3000:1, and (3) increasing the array density to > $10^9$ wires/cm$^2$. The primary objective of this project is to fabricate and test the thermoelectric properties of arrays of BiSb nanowires.

## Accomplishments

- Electrochemical Mass Transport Issues for BiSb Deposition:

Since nanowire diameters and lengths are controlled by the nanopore diameter depth of the anodized aluminum oxide (AAO) templates, we addressed how mass transport affects the BiSb nanowire composition during high-aspect ratio deposition compared to similar operating parameters designed for films of the same composition on flat surfaces. The reducing currents used to electrodeposit material in pores are a function of the driving potential, the product of three mass-transport modes that act resistively to current flow. Diffusion, convection, and ionic migration each limit the Nernst potentials needed to generate charge transfer. We eliminate ionic migration resistance by using a highly conductive supporting electrolyte. Convective resistance is eliminated since agitation in pores < 200 µm is negligible. This leaves diffusion-induced concentration gradients as the main mass transport issue.

We studied composition and mass-transport issues using a simple rotating disk electrode (RDE) and a Hull cell to perform the experiments. Our experiments demonstrated that maintaining the BiSb composition in nanopores due to diffusion is NOT a major issue. However, major current limitations can come from the rate of double layer charging in the interphase. Indeed, as the potential increases negatively from thermodynamic equilibrium, the total cathodic current increases linearly for both $Bi^{3+}$ and $Sb^{3+}$, with Bi depositing at a greater ratio than Sb.

The Hull cell experiments show that a nonuniform field flux, producing changes in the driving potential, controls both film composition and morphology. Since both Bi and Sb are highly dependent on the applied potential, the ionic solution composition controls the final composition. Taken together, the RDE and Hull cell results demonstrate that compositional uniformity in high-aspect ratio nanowires should not be a problem.

- Concept Demonstration:

This involves two main areas. The first is fabricating AAO templates with controlled nanopore sizes and densities over great lengths. The second is growing vertically aligned BiSb nanowires in the templates; we accomplished this in collaboration with Angelica Stacy at the University of California, Berkeley.

We formed AAO nanopore templates by electrochemically anodizing radio frequency (RF)-sputter deposited Nd-doped Al films on Si substrates, with thickness determining nanopore depths. (Nd-doping provides mirror-smooth Al films with less surface oxidation and some stress relief.) Ultimately, we desire nanopore depths approaching 100 µm. The Al films delaminate if their residual stress is large, rendering the templates useless. We studied methods to reduce the film stress during deposition and developed procedures to make it negligible. We demonstrated stress-free 10 µm thick films that should not spall at greater thicknesses.

In collaboration with Professor Stacy, we demonstrated BiSb nanowires with lengths approaching 60 µm in AAO from foils, and we are now transferring the process to AAO templates on substrate surfaces; this provides a good thermal contact between the thermoelectric nanowires and the substrate to be cooled.

- Atomistic Modeling to Determine Impact of Grain Boundaries and Nanowire Contacts:

BiSb nanowires are polycrystalline with grains smaller than the phonon mean-free path, indicating that grain boundary (GB) and free surface contact scattering may dominate mechanisms for heat flow resistance. We developed new tools to model the influence of interfaces on phonon transport as a step toward predicting the thermal transport properties of synthesized nanowires. We are able to use realistic models of GB structures in materials. This algorithm permits us to form boundaries with varying twist and degree of disorder. We also developed the ability to model phonon wavepackets impinging upon boundaries. In addition, we developed analysis tools to extract relevant information such as transmitted/reflected energy and frequency. Trial simulations on an ideal interface agreed with published results, validating our implementation. In collaboration with Patrick Schelling at the University of Central Florida, we are studying the effect on phonon transport of disorder in GB structures for one twist angle to compare with existing results on an ideal interface. This will bring us the ability to model nonnormal phonon incidence on GBs. This is necessary to address the full phase space relevant to thermal transport in BiSb nanowires and other nanostructures.

## Other Communications

C.J. Kimmer and E.B. Webb III, “Phonon Scattering by Molecular Dynamics: Temperature Dependence and Effect of Structural Disorder,” presented at the APS March Meeting, Los Angeles, CA, March 2005.

# Advancement in Thermal Interface Materials for Future High Performance Electronic Applications

*84320*
*M. J. Rightley, J. A. Galloway, C. C. Wong, D. L. Huber*

## Abstract

As electronic assemblies become more compact and need increased processing bandwidth, the escalating thermal energy becomes more difficult to manage. The major limitation is nonmetallic joining using poor thermal interface materials (TIMs). The interfacial thermal conductivity of an adhesive is the major loss mechanism and normally accounts for an order of magnitude loss in conductivity per equivalent thickness. The next generation TIM demands a sophisticated understanding of material and surface sciences, heat transport at submicron scales, and the manufacturing processes used in the packaging of microelectronics and other target applications. Only when the relationships between bond line manufacturing processes, structure, and contact resistance are well understood on a fundamental level will it be possible to advance the development of miniaturized microsystems.

We propose to use thermal and squeeze-flow modeling to formulate TIMs that incorporate nanoscience concepts. Understanding the thermal behavior of bond lines will allow us to focus on the interfacial contact region. We will use tools developed in a previous LDRD project to develop process guidelines for optimum surface wetting (void free).

Careful study of the thermal transport across these interfaces will not only provide greatly augmented heat transfer paths, but will allow the formulation of very high resistance interfaces for total thermal isolation of circuits. As an example, this will allow the integration of systems that exhibit multiple operational temperatures, such as cryogenically cooled detectors.

## Accomplishments

We reviewed and assessed the existing thermal conductivity models of adhesive polymers. We identified key phonon scattering mechanisms of amorphous polymers. We developed a subgrid physics model that can calculate thermal conductivity of amorphous polymers. Assessment of the model and its comparison with experimental data are in progress.

We synthesized silver nanoparticles ranging in size from 5 to 10 nm and established protocols to disperse purified nanoparticles into epoxies. The nanoparticles fully disperse in the epoxy matrix. Evaluation of the nanocomposite revealed no additional benefit to adding the current version of silver nanoparticles; however, further iterations are in progress. We also developed a synthesis for 10 nm magnetite particles to explore their use as magnetic field structured fillers. These nanoparticles can be dispersed, then structured as is, or can be treated with a more conductive metal coating prior to incorporation into the epoxy.

We developed the ability to control and monitor the bondline assembly process. We can perform an evaluation of bondline thermal properties and microstructure for silver-flake filled thermal interface material. By monitoring force, varying squeeze rate, and observing final bondline thickness, we discovered that slow squeeze rates lead to thicker bondlines and reduce thermal performance. During the squeeze flow, the filler flakes become oriented, and the effective thermal conductivity appears to increase at smaller bondline thicknesses. We measured thermal diffusivity by laser flash, and microstructure by optical and electron microscopy.

## Other Communications

J.A. Emerson, J.A. Galloway, D.F. Rae, and M.J. Rightley, “Thermal Interface Materials Advancements for ‘Beating the Heat’ in Microelectronics,” presented and in *Proceedings of the SAMPE 2005, Technical Conference Proceedings: Advancing Materials in the Global Economy—Applications, Emerging Markets and Evolving Technologies*, Long Beach, CA, May 2005, vol. 6, pp. 1–9.

J.A. Emerson, M.J. Rightley, J.A. Galloway, D.F. Rae, D.L. Huber, and E.J. Cotts, “Minimizing the Bondline Thermal Resistance in Thermal Interface Materials Without Affecting Reliability,” presented and in *Proceedings of the 11th International Workshop on Thermal Investigations of ICs and Systems THERMINIC 2005*, Belgirate, Lake Maggiore, Italy, 2005, vol. 11, pp. 106–111.

D.F. Rae, M.J. Rightley, J.A. Emerson, J.A. Galloway, D.L. Huber, and E.J. Cotts, “Thermal Interface Control: Thermal Performance and Structural Correlations for Microscale Composites with Dispersed Nanoscale Filler Material,” presented at the 28th International Thermal Conductivity Conference and 16th International Thermal Expansion Symposium, St. Andrews by-the-Sea, New Brunswick, Canada, June 2005.

D.F. Rae, M.J. Rightley, J.A. Emerson, J.A. Galloway, D.L. Huber, and E.J. Cotts, “Enhanced Thermal Bondlines: Nanocomposite Thermal Interface Material and the Minimization of Bondline Thermal Resistance,” presented at the Materials Science & Technology 2005 Conference, Pittsburgh, PA, September 2005.

# Intrinsic Code Verification Signature Generation

*92368*
*B. D. Schoeneman*

## Abstract

A purpose of intrinsic code verification (ICV) signature generation is to authenticate, with very high confidence, that the firmware/software/data (code) embedded in a device is the original software installed in the device and that the code has not been changed. Prior to engaging in this process, the original software can go through a vulnerability assessment by a trusted agent. The original software package then becomes the reference package for all subsequent installations, and the package can be protected at the same level as the keys used for authentication and verification of the code space.

To derive the high confidence indication that the embedded code has not been altered, a hashing algorithm is run on the entire contents of memory to derive a signature. The authentication algorithm used to generate the hash signature of the embedded device's memory contents actually resides within the executable code, hence the term "intrinsic." To begin the hashing process, a key, a seed (can be random), and a selected insertion point for the seed (can be random) are passed to the embedded device in a message, from a host, requesting code verification. The key is used by the authentication algorithm to generate a signature of the contents of the device's memory. The seed is inserted into the authentication process at the memory location specified by the selected insertion point. The final result is a multibyte signature of the varied contents of the embedded device's memory. The signature is returned to the host for verification. The authentication process is performed on the protected reference code package, including the same bit values in the embedded device's remnant memory, residing on the host device. Verification is established by comparing the signatures of the two hashing processes, resulting in a pass or fail indication. This entire process can be repeated to establish increasingly higher confidence (confidence increases as power of number of times ICV is repeated) in the verification result.

## Accomplishments

To begin our investigation into the viability of the concept for ICV signature generation, we chose a standard authentication algorithm. We implemented the HMAC (keyed-hash message authentication code) utilizing SHA-1 (secure hash standard) in "C" code. We tested the implementation of these algorithms against the FIPS-180 (federal information processing standard) standard test cases to confirm accurate operation. We then incorporated the resulting C code implementation of this HMAC w/SHA-1 into supporting C code to create two modules for test and evaluation of the ICV concept. We wrote one of these modules for, and installed it into, a target embedded platform. For the purposes of this project, this platform was the T-1A fiber optic loop seal used for item monitoring in long term material storage applications. We wrote the second software module as a DOS application and installed it on a standard personal computer.

The purpose of the code embedded into the T-1A was to simulate a fielded security device that needs to be "trusted" in its production of verifiable information/data. If the embedded software in the security device were maliciously altered, it could circumvent the security provided by the seal, in this scenario, and provide false indications of its status. We intend the ICV to detect this alteration with very high confidence. The operation of ICV in this scenario would command the security device to run the HMAC with SHA-1 on its internal binary memory using a preexisting internal private key (programmed into the device at some time prior to this process) and a randomly introduced and randomly generated seed, both provided by the command at the time of the command to produce a 20 byte hash signature. The hash signature is a representation of the contents of program memory with the seed insertion. The entire process took ~12 seconds to hash 32,768 bytes of memory. The 20 byte HMAC was then output from the device through its serial port and captured for reference. It must be noted that, for the purposes of this project, we hard coded the private key, manually entered the seeds, and manually chose the locations for insertion of the seeds. Additionally, we did not randomize unused memory padding; it should be randomized for actual application.

The code we installed on the personal computer performed the same function as the embedded system,

with the major exception that the ICV was performed on a reference image of the embedded code memory. We manually compared the resultant 20 byte HMAC to the HMAC result from the embedded device. A match indicated that the two processes authenticated the same memory contents with the same seeds inserted into the same locations; in other words, the embedded code had not been altered.

We conducted additional testing to confirm that multiple builds (compilation and linking) of the same source code for the embedded device would produce consistent outputs. This was confirmed to be correct. We also performed testing to induce very slight changes in the embedded source code without changing the reference memory image. The ICV process of validation successfully detected all of these alterations.

At this point, we consider the fundamental ICV concept to be viable for the detection of surreptitious alteration of trusted embedded firmware. Additional associated areas still need to be investigated before the ICV process can actually be implemented and utilized as described. Some areas of concern are: empty space padding and off chip memory, lossless code compression, implementation of SHA-256 instead of SHA-1, the implementation of actual commands, and the evaluation of random seeds and locations.

# Developing a Viable SCADA IO System Architecture

*93032*

*L. G. Pierson, J. E. Stamp, C. H. Villamarin, D. L. Harris, D. P. Duggan, R. R. Olsberg, B. D. Kucera*

## Abstract

This project developed and documented a reference architecture for an integrated information operations (IO) system that will function with supervisory control and data acquisition (SCADA)-specific technology.

Protecting the nation in the cyber arms race necessitates a strategic approach to information superiority. Previous research has provided insight into national strategic guidance for information superiority. In this project, we began to apply systems engineering approaches to strategic information surety systems and concepts that underpin national security needs.

We explored the use of a specific architecture for detection of malicious activity, instantiating protections, and for damage assessment in strategic information surety systems. We began to lay a foundation for effective protection of SCADA systems from adversaries using IO techniques.

## Accomplishments

We developed and documented a reference architecture that facilitates system and design analysis and provides a basis for prototype development. We conducted network protection experiments to show the viability of the architecture to be robust enough to accommodate changing threats, concepts of operation, and the evolution of information technologies. We began the top level design of prototype systems consistent with the reference architecture in order to demonstrate viability of this architecture to enhance information surety. While construction of these prototypes is beyond the scope of this project, we conducted certain laboratory experiments to ascertain feasibility and scalability of the concepts. These experiments validated the usefulness of the proposed architecture to protect energy distribution infrastructure against information based attacks.

# Improved Radar Pulse Detector for Radar Responsive Tags

*93033*

*R. C. Ormesher, K. A. Sandquist, S. M. Patel*

## Abstract

The purpose of this project was to investigate a new method of detecting chirped radar pulses. Development of a more sensitive tag pulse detector will allow the radar-to-tag downlink to function in scenarios requiring greater standoff ranges or foliage penetration.

The first achievement is a Monte Carlo simulation that provides a performance comparison of the new chirp-pulse detector and the standard (currently used) video detector. The new detector promises to be more than an order of magnitude more sensitive than the existing detector. The second achievement of this project is a hardware prototype of the new detector. We developed this prototype to verify feasibility and performance of the new design.

## Accomplishments

This effort focused on two primary areas: (1) simulating and analyzing the proposed concept and (2) developing a hardware prototype of the new detector. First, we developed Monte Carlo simulations to verify both the proposed concept and previous theoretical analyses. We implemented this simulation in Matlab, partially parallelized, and executed it on several Linux-based PCs (personal computers). The simulation provided a curve of probability of detection versus signal-to-noise ratio (SNR), given a particular probability of false alarm for the new detection method. We then compared the results of the simulation to performance estimates for a standard video detector. We approximated the probability of detection for the standard video detector using methods that are well known and found in text books. The comparison results show that the new method of signal detection provides an improvement of approximately 13.5 dB relative to the standard video detector. Since many iterations were necessary to achieve the desired level of accuracy, the simulation demanded several days of processing time to complete for a fixed set of operating parameters. Therefore, we developed a new mathematical model to reduce the processing time. Based on the new mathematical model, we implemented a new Monte Carlo simulation in Matlab and reduced processing time from days to hours. When comparing the results of the new simulation to the original simulation, we found a discrepancy of 3 dB. We have not yet investigated this discrepancy.

Second, we developed a hardware prototype of the detector design using commercial-off-the-self radio frequency (RF) components. We designed the detector prototype to operate at 1 GHz and with an input bandwidth of up to 500 MHz. We assembled the detector in the laboratory and are using it to verify the Monte Carlo simulation results. A first level bench test shows that the detector is functional and operating as expected. We designed the 1 GHz input into the detector to align with the intermediate frequency (IF) center frequency of an existing RF radar responsive tag. This will allow us to further characterize the detector, both in the laboratory and in field tests, using an existing tag and existing compatible synthetic aperture radars that employ wide band chirp transmit pulses.

In summary, using Monte Carlo analysis, we demonstrated that the new method of chirp-pulse detection provides an improvement of approximate 10 to 13 dB relative to the standard video detector. We developed prototype hardware and used it to demonstrate that the new chirp detector operates as expected. We can use the prototype hardware to further characterize the detector in future laboratory and field tests.

# Nonproliferation Chem/Bio Scenarios

*93034*

*N. B. Barnett, M. Finley, K. Silver, D. Evanko, K. E. Horak, J. M. Gaudioso*

## Abstract

The objective of this project is to develop and analyze scenarios that would lead to an adversary's ability to acquire and use a biological weapon. These scenarios have not been described for the field of biological weapons nonproliferation (BWNP). We will consider four main factors in the design of nonproliferation and assessments (NP&A) scenarios: adversaries (state and nonstate actors), acquisition, development, and dissemination of a biological weapon. An analysis of adversaries requires consideration of their possible objectives, which may include asymmetric strength (coercive, retaliatory) through terror or genocide, invigoration of a factional support base, assassination, territory denial, and incapacitation of opponents on the battlefield. Adversaries must first acquire suitable biological materials through one of several plausible pathways, such as theft from a legitimate facility, theft during transport, isolation from natural sources, acquisition from a state program, or genetic engineering. Once acquired, the adversary must produce an appropriate quantity in a suitable form. Testing, storage, and quality assurance may also be components of the development stage. Once a biological weapon has been developed, there are a broad range of options for dissemination, from traditional state-based weaponry to commercially available crop dusters or envelopes in the mail.

The successful completion of this project will result in the identification of scenarios that pose the greatest risk to national security and highlight appropriate countermeasures to mitigate these risks. Such countermeasures may include intelligence measures, laboratory biosecurity, disease outbreak surveillance, biodetectors, and consequence management tools. By identifying technologies and policies that can be developed to prevent an event from occurring, these scenarios could not only help focus and prioritize US biological weapons prevention efforts overseas, but may also help the US biodefense community re-evaluate the countermeasures developed to respond to bioterrorism in the US.

## Accomplishments

We initially intended to develop a preliminary nonproliferation scenario to demonstrate the utility of NP&A scenarios in prioritizing BWNP activities worldwide. Instead, we devoted more effort to the acquisition pathways, specifically culture collection locations for the entire set of pathogens currently under review: *Variola major* (smallpox), *Bacillus anthracis* (anthrax), *Yersinia pestis* (plague), *Foot-and-mouth disease virus* (FMDV), *Vesicular stomatitis virus* (exotic), *Clostridium botulinum* (botulin toxin), avian influenza, Venezuelan equine encephalomyelitis (VEE/EEE), ricin, Staphylococcal enterotoxins (Staph), and *Salmonella typhi*. We also studied the current legislative restrictions for these pathogens. These activities moved the project forward in establishing the underlying conditions that may enhance or reduce the risks associated with these pathogens being acquired illicitly. Understanding the acquisition pathways prior to establishing an end to end scenario appears to be a better path forward than the one originally proposed. The technical details are important to establish prior to attempting to develop an overall scenario.

Environmental sources of biological agents include endemic areas and outbreak sites. News of recent outbreaks can be obtained through surveillance data collected by a number of public and agricultural health organizations; we began evaluating such websites.

Thus far, we found that we can obtain *Bacillus anthracis*, *Clostridium botulinum*, *Francisella tularensis*, and various species of *Staphylococcus* and *Salmonella* from a number of culture collections; however, these agents require special approval prior to purchase. We also identified a number of diagnostic laboratories in Southeast Asia that test samples for *Bacillus anthracis*, highly pathogenic avian influenza, *Francisella tularensis*, *Yersinia pestis*, *Salmonella typhi*, and *Salmonella typhimurium*.

We have FMDV outbreak data on a country wide basis from 1999 to 2005, and, as a result, we can determine where FMDV is likely to be endemic and where recent outbreaks have occurred. We also used the National Library of Medicine (PubMed) to identify research institutes and scientists studying FMDV.

We are conducting research to determine both the mandatory and voluntary requirements for obtaining pathogens from legitimate facilities. This effort focuses primarily on possession and acquisition requirements: transfer (interstate), import, export, re-export (e.g., US Code of Federal Register) laws, and international agreements (e.g., Australia Group, est. 1985) enacted to prevent the illegal acquisition of pathogens and toxins. Additionally, we will identify current exemptions to regulations and nonparticipants of international agreements. We identified national legislation for the US, United Kingdom, and Canada. We identified summaries of international legislation on a country-by-country basis and are working to acquire them from external organizations.

We conducted database development work to establish country specific information related to biofacilities, biotechnologies, disease surveillance, endemic diseases, regulations, research centers, and the political climate. We also developed facility tables containing information on activities, biosafety, pathogens, security, and Sandia's assessments. We created a prototype of the database and conducted a demonstration of its functionality.

# Laser Propagation Analysis

*93035*

*B. W. Atherton, M. T. Valley, W. J. Tedeschi, D. J. Armstrong, W. C. Sweatt, A. V. Smith*

## Abstract

Pulsed lasers can significantly impact our nation's monitoring capabilities. We will deliver three analyses: (1) we will quantify the feasibility and timeline, (2) provide additional design information, and (3) plan for experimental validation and refinement of the first two items. This work created a cadre of Sandians that can analyze new issues in this area, advise our leaders about them, and establish relationships with other federal laboratories. We believe that a laser flux as low as a few mJ/cm$^2$ can pose a significant threat to monitoring systems; this appears to be well within the deliverable energy ranges of explosively driven iodine lasers, modern projection systems, and atmospheric compensation systems. A single shot device will likely use a pre-shot tracking system. Removal of atmospheric disturbances is critical to the energy density delivered on target. The tracking system is likely also used as a probe to compensate for atmospheric distortions, employing either adaptive optics or other means.

The project has four distinct objectives: (1) analyze the source architectures and characteristics (beam quality, energy, pulse duration/shape, etc.), (2) determine the design approach limitations of the projection system, (3) understand the limitations and compensation techniques of propagation, and (4) identify beam characteristics at range.

We will need the initial analysis of the source as a starting point for the remaining tasks. The initial analysis includes literature searches of published material, as well as discussions with outside organizations (US Air Force, national laboratories, etc.). We developed computer simulations of the laser sources with differing configurations, various projection approaches, and propagation and atmospheric compensation techniques to increase understanding of effects on the beam parameters at range.

## Accomplishments

This project assessed damage mechanisms, atmospheric propagation effects, and different mechanisms to mitigate the atmospheric effects. We then established the requirements and limitations for the source. The damage mechanisms are summarized below:

(1) There are effects that can occur when a laser engages a solid target, including heating, melting, vaporization, and momentum transfer. Heating begins as soon as laser light is absorbed at a target's surface. The other effects begin at progressively higher intensities.

(2) The reaction force imparted to a surface by the vapor as it blows off produces a pressure. This pressure can serve to damage the target, provided the impulse is great enough to rupture the target's surface.

(3) The advantage of target erosion (melting or vaporization) as a damage mechanism is that the effects are well understood and it requires lower intensities (W/cm$^2$). Mechanical damage by impulse delivery requires less fluence (J/cm$^2$), but is more difficult to characterize and requires higher intensities and shorter pulse widths. Plasmas are more likely to occur and influence the interaction at these higher intensities.

To determine the atmospheric effects, we developed a code using a Hufnagel–Valley atmospheric turbulence model (daytime, inland sites). We chose a scenario target located 80 km slant range with an altitude of 15 km. We then looked at ways to help get the energy from the source to the target. The first method employed a noncooperative target that required energies on the order of damage to overcome the spontaneous emission modes of the explosively driven laser. The second method we explored used relay mirrors that employed a cooperative beacon. This greatly improved our energy density on target. Finally, we employed brute force, using the largest energy source with a flat director mirror that could track the target; this also gave reasonable energy density on target.

Next, we looked at the potential sources. We first looked at the highest energy explosive iodine laser. This gave a pulse with very poor beam quality. The second source we considered was a source with phase conjugation. Limitations with the second source included hard to control beam overlap in the phase conjugation material, and a very small operating range. The last source we analyzed was a poor quality high energy beam with nonlinear amplification from either a Raman or nonlinear crystal. There are several plausible ways to use nonlinear optics to clean up the beam of a

high energy pulsed iodine laser. Competition among nonlinear effects makes it difficult to precisely determine the limits of efficiency and beam quality using these effects without performing experiments.

# Vulnerability Assessment Modeling of Extended Detection Systems

*93349*

*M. K. Snell, J. L. Darby, C. L. Nelson*

## Abstract

Current vulnerability assessment software tools do not have established methodologies for modeling extended detection systems (thermal, radar, etc.). In this project, we revised current path event timeline models to reflect new criteria for using extended detection, such as determining temper and intent. New systems also provide opportunities for supporting delay systems and for vectoring response forces to where such forces can best be used; we identified some of these opportunities. As a result of this work, we identified ways to better test such systems and to use existing computer codes such as ATLAS (Advanced Timeline Analysis Software) to represent several of the major vulnerability issues that could potentially arise from implementing extended detection systems. These new modeling approaches show promise in demonstrating that extended detection systems are more capable than traditional models give them credit for.

## Accomplishments

We developed a list of general criteria for extended detection systems that support not only detection, but also address delay and response objectives. We then developed threat scenarios that consider generic ground (foot, vehicle) and air attacks. Analysis models for extended detection against each of these attacks include multiple timelines, where each timeline is based on a combination of two factors: (1) what is known up to a given point in time about a possible intrusion (e.g., number of adversaries, presence of weapons) and (2) what objectives the security force is attempting to accomplish at that point (e.g., sensing, assessing, establishing intent, imposing delays, and conducting interruption and neutralization). We then examined these timelines to identify testing needs for components of extended detection systems.

As a result of these activities, we identified the practical detection point algorithm in the ATLAS vulnerability assessment model as one analytical approach for examining the performance of such systems. We developed a method to use the new system's effectiveness formula for extended detection systems. We also documented recommendations for improving current timeline models to model modern alarm systems.

## Refereed

D.G. Adams, M.K. Snell, M.W. Green, and D.A. Pritchard, "Between Detection and Neutralization," presented at the 39th IEEE International Carnahan Conference on Security Technology, Las Palmas, Spain, October 2005.

# Feasibility Study on Using Department of Defense Tech Demo Technologies

*94253*

*M. R. Ackermann*

## Abstract

The purpose of this project was to assess the feasibility of merging technologies from programs belonging to the Department of Defense (DoD) with unique Sandia technologies to address shortfalls in Department of Energy (DOE) mission space for nonproliferation monitoring and assessment. We made some progress, but we did not meet all objectives.

## Accomplishments

The concept was to assess the feasibility of combining technologies from DoD programs with technologies available at Sandia to enhance the ability of DOE to perform nonproliferation monitoring activities. This feasibility assessment included both an assessment of technologies presently covered by DoD programs and an assessment of the ability to merge technologies to support DOE missions and possibly other DoD and intelligence community organizations.

The project plan included the following tasks:

(1) Establish a team of Sandians with the correct security clearances and technical expertise and meet with external organizations to establish rules of engagement.

(2) Identify external technologies that may be applied to known DOE and intelligence agency missions supported by Sandia, and identify Sandia technologies and capabilities that are synergistic with those available from DoD.

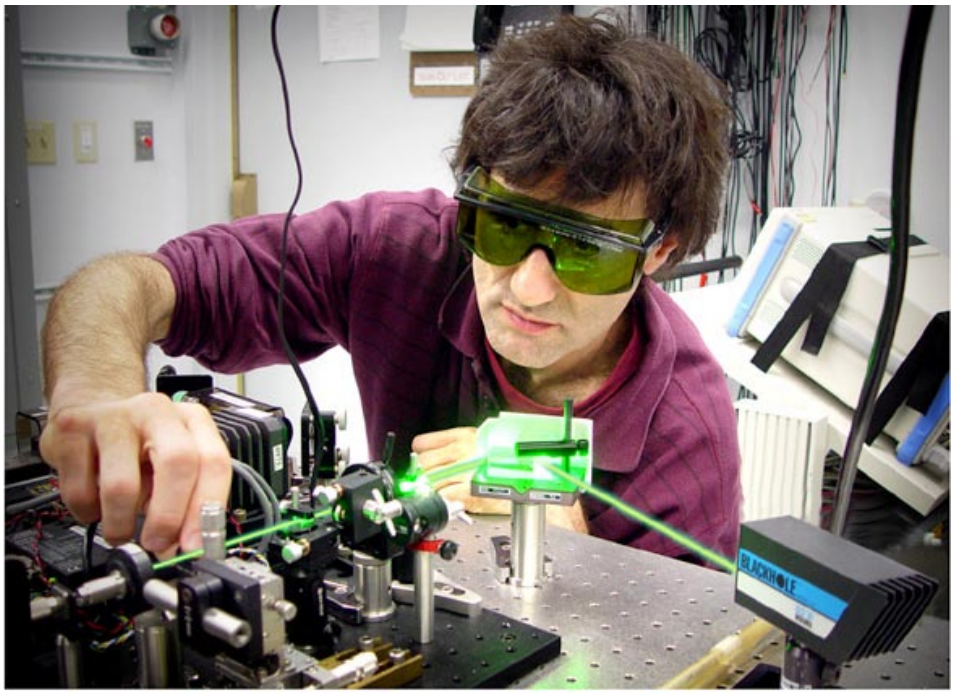
We were successful at contacting DoD program managers and generating interest in the concept. Our initial assessment that there would be a natural and compelling reason to attempt to seek synergistic technologies appears to be correct. We examined technical monitoring areas and mission requirements, where Sandia supports either DOE or external agencies, and found several examples where technology from at least one of the DoD programs could be combined with Sandia developed technology to address new monitoring missions. We conveyed our interest in these areas to the appropriate program offices. We did encounter a problem, inherent in this type of work, in trying to coordinate information exchange between dissimilar but carefully controlled programs.

In summary, we achieved moderate, but not complete, success. We established that there exists high level interest in combining capabilities. We also established that there are reasons for Sandia involvement and benefits to Sandia, the DOE, and our traditional DoD and work-for-others customers.

**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. Grand Challenge investments are often system-level projects conducted across multiple disciplines. 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.

Grand Challenges seek solutions to national critical problems. For example, a significant national energy challenge is developing a comprehensive systems approach to nuclear energy, including advanced reactor design surety features, fuel-cycle proliferation resistance, innovative reprocessing options, waste disposition, and accountability mechanisms.

The Grand Challenge investment area currently includes nine new and ongoing projects that are addressing national problems in fuel cell research, cognitive science, physical security, border defense, advanced fusion, sensor networks, underground characterization and defeat of targets, global response hypersonic vehicles, and high power lasers.



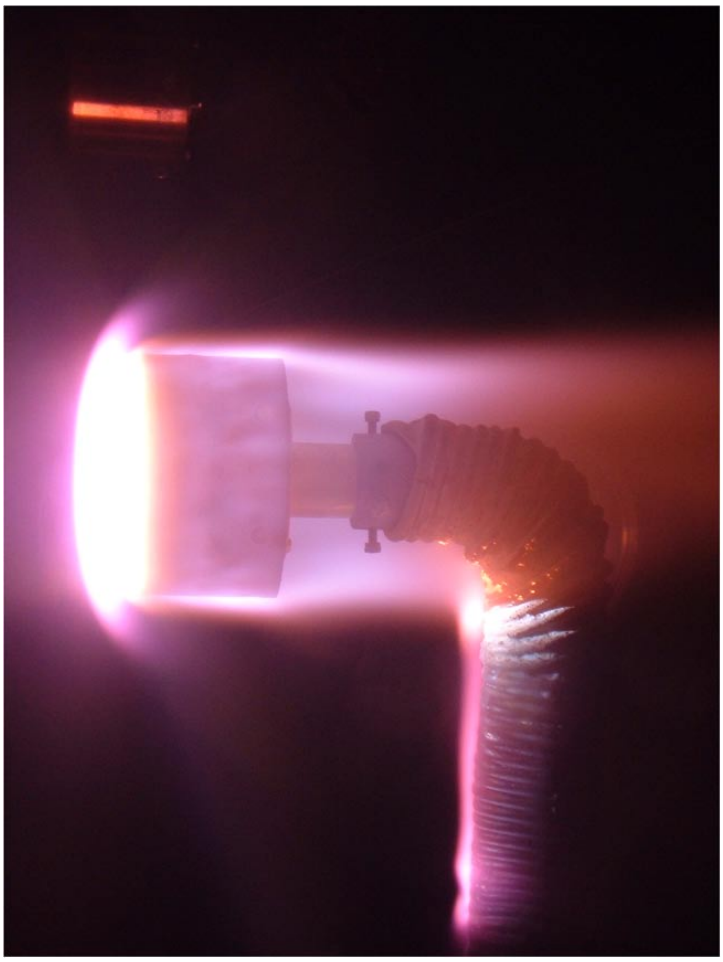
**81752 "Integrated Fiber Lasers for Efficient High Power Generation"**

Sandia researcher Dahv Kliner and his team developed a bend-loss-induced mode filtering method that allows the use of large core diameter fibers while maintaining high beam quality.

More specifically, project **81752 "Integrated Fiber Lasers for Efficient High Power Generation"** seeks to revolutionize the field of high power lasers through the use of disruptive new technologies, such as rare earth-doped fiber lasers, that will overcome the limitations of current systems that are bulky, fragile, inefficient, and have poor beam quality.

The versatile new laser technology will be capable of simultaneously meeting the stringent combination of optical and physical requirements of practical high power lasers. The success of the project thus far is primarily based on breakthroughs in radical power scaling of the rare earth-doped fiber lasers. Researchers are making leading edge advancements in rare earth-doped fibers and fiber amplifiers, pump diodes, microlaser seed sources, waveguide and laser/amplifier modeling, and system engineering and testing. Advancements thus far enable the peak and average power of fiber sources to be increased by at least two orders of magnitude without sacrificing beam quality or efficiency. In addition, the bend-loss technique has been adopted worldwide and is now the standard for power scaling. Researchers are named in five patents that enable the fiber laser technology that will facilitate compact, rugged high power lasers for military, intelligence, homeland security, and nuclear weapons surety applications.

Grand Challenge project **55086 "Prompt Global Response**," will also find application across multiple national security areas. Researchers are working to develop advanced technologies and integrated capabilities for hypersonic vehicles as long range, unmanned precision delivery platforms. Areas of research and development include materials development, electronics, engineering, radar, physics, modeling, and flight computer architecture. Researchers developed engineering designs of a boost-glide vehicle, and new global positioning algorithms and receiver software. In FY 2005, they achieved advancements in navigation, guidance and control, and vehicle thermal protection. Researchers also recently developed a new model for thermal and pressure response, and simulated the effects of trajectory tailoring, velocity at reentry, vehicle weight, lift-to-drag ratio, and booster and hand-off altitude. The project has already received favorable attention from organizations such as the Defense Advanced Research Projects Agency, the National Aeronautics and Space Administration, and the US Strategic Command.



**55086 "Prompt Global Response"**

Arc jet test of carbon-carbon oxidation protection material developed in the Prompt Global Response thermal protection task.

# Bio MicroFuelCell

*41193*

*C. A. Apblett, B. A. Simmons, P. L. Dolan, S. M. Brozik, D. Ingersoll, G. A. Roberts, J. C. Harper, M. Manginell*

## Abstract

A Bio MicroFuelCell would provide power by harvesting energy from the environment, making it possible to realize unprecedented lifetimes in a variety of self-sufficient microsystems. Autonomous, distributed power systems are required for cooperative microsensor networks being developed for a number of applications, including battlefield and border management, surveillance, monitoring, and intelligence gathering scenarios. This Grand Challenge project embodies the first steps toward the long-range Bio MicroFuelCell vision, and lays the groundwork for a robust, multidisciplinary research and development program.

This year we focused first on the development of the genetic engineering capability to grow and purify modified enzymes in sufficient quantities for use in a fuel cell environment. Next, we focused on developing a means to immobilize both the enzymes and the mediators on an electrode that could be incorporated into a standard fuel cell configuration. We built a fuel cell made with phenazine mediated glucose oxidase anodes. It produced about 250 µW of power at room temperature using only a 50 mM concentration of glucose. Adjusted for concentration, this power is greater than the power developed from even the noble metal group catalysts, and represents a significant step forward in power production for enzymatic cells.

## Accomplishments

We demonstrated the ability to genetically modify proteins based upon glucose oxidase to increase the activity of the protein for the oxidation of glucose by a factor of three. The method of determining this activity was originally based upon optical spectroscopy, was not particularly reproducible, and introduced large errors into the calculation of the rate constants for the oxidation, which limited the amount of kinetic data that could be developed. We developed an electrochemical assay that directly measured glucose oxidation, and used it both to verify the activity of the modified organisms and to calculate the relevant kinetic parameters of the catalysis.

Once these new techniques were in place, we developed protocols for the expression of the new protein using an *Aspergillus niger* host. The expression of the protein allowed for large quantities of protein to be generated and secreted through the cell wall. We developed purification protocols to separate the protein of interest from the other excreted proteins. Separations based upon nickel-nitrilotriacetic acid (nickel-NTA) columns binding to a gene modified histadine tag on the surface of the protein allowed for purification of the protein. Subsequent recovery of the protein from the separation column allowed for a pure enzyme recovery. Although the protocols developed resulted in a low protein recovery, Western blots of the recovered protein demonstrated that it was very pure, and further development of the protocols can be expected to increase the recovery percentages.

The enzymes were successfully immobilized onto carbon electrodes using a diaminoquinone linkage. This linkage appears to stabilize the enzyme on the electrode surface, resulting in much higher currents than previously observed. Using phenazine based mediators, we observed currents in the multiple mA range for a single anode in 50 mM glucose. We were unable to achieve success with the immobilization of the mediator, however, and an alkyl modified phenazine mediator molecule synthesized with an amino terminator for covalent bonding did not exhibit mediating behavior. Nonetheless, using phenazine in the fuel solution to provide mediation, a fuel cell using covalently linked enzymes produced 250 µW of power, which was greater than a similarly constructed fuel cell using noble metal anode catalysts.

# Augmented Cognition: Next Generation Intelligent Systems

*41194*

*J. S. Wagner, D. H. Hart, E. P. Parker, B. E. Hart, R. J. Anderson, A. E. Speed, A. Backer, M. L. Bernard, N. M. Berry, K. W. Larson, P. G. Xavier, J. C. Forsythe, C. M. Pancerella, R. L. Waymire, J. W. Wirsbinski, D. E. Small, C. W. Wilson, C. L. Lewis, M. R. Muguira, A. I. Gonzales*

## Abstract

The objective of this project was to develop innovative solutions to augment the ability of humans to recognize hidden, but meaningful, patterns within massive quantities of data. To address this goal, the project advanced the state of the art in perceptive and cognitive systems, enabling systems to be created that integrated these technical capabilities. We advanced perceptive systems through the development of capabilities that integrated simultaneous location mapping (SLAM) and scale-invariant feature transform (SIFT) algorithms to provide object recognition in a manner that dynamically tracks objects with respect to the environmental surroundings. We advanced cognitive systems through the development of a computational framework for modeling human context understanding and techniques for automated knowledge capture that allow a cognitive model of a specific individual to be acquired through observation of their behavior. We created a systems engineering infrastructure for integration of perceptive and cognitive components, and implemented an agent architecture that provides agent encapsulation of the cognitive framework. The project concluded with a successful integrated demonstration that combined perceptive and cognitive systems to augment human interpretation of adversary tactical behavior in a heavily sensored facility.

## Accomplishments

We achieved numerous accomplishments in five major areas.

(1) Perceptive Systems (PS) Visual Object Recognition

(a) We created three-dimensional object recognition algorithms that support spatially informed perception and SLAM.

(b) We created a spatial interface with the cognitive system that enables spatial memory, with the broader objective of cognitive system embodiment.

(2) Cognitive Systems (CS) Context Based Understanding

(a) We developed an accurate computer model of the cognitive processes in which an individual applies their unique knowledge and experience to interpret data and events.

(b) We developed techniques that enable an accurate model of a specific individual's knowledge of a task or domain to be acquired, using data generated during routine activities.

(c) We developed augmentation technologies based upon our ability to model the cognitive processes of a specific individual that produce superior human performance.

(3) Extensible Computational Systems Engineering

(a) We developed the main components of a practical implementation of the upgraded cognitive framework that will enable integration with PS and other future NGIS components.

(b) We developed a multi-agent architecture that incorporates agents with embedded cognitive frameworks.

(c) We extended the core architecture to enable a distributed, scalable NGIS system integrating cognition with perception.

(4) Integrated Demonstration

We conducted an experiment that will provide a quantitative assessment of integrated PS and CS technologies to enhance the situational awareness and performance of physical security forces.

(5) Cognitive Sciences and Technologies (CS&T) Roadmap

We assessed critical needs for Sandia's strategic managements units and created a technical roadmap for CS&T at Sandia to position the laboratories to contribute new capabilities in this area in support of our national security mission.

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# TALON

*50065*

*F. J. Oppel III, M. B. Murphy, J. R. Yoder, K. T. Gee, D. D. Spencer, J. B. Woodard, F. T. Mendenhall, D. A. Wiegandt, J. P. Anthes, M. H. Johnson, M. J. Carr, R. M. Axline, C. C. Mitchell, L. G. Pierson*

## Abstract

The fundamental hypothesis of this project is that sensor networks provide a robust system solution for many problem classes from intelligence collection to military force protection, Department of Energy asset protection, Department of Homeland Security ports and border security, and countless others. To focus the effort, we chose to consider the problem of detection, classification, and tracking of a diverse set of mobile ground targets using heterogeneous unattended ground sensing, adaptive communications, and long-lived efficient network and sensor management. It is expected that sensor networks will lead to significant improvement in capability over existing solutions. We tested this hypothesis by exploring the collaboration and multisensor data fusion enabled by local networking to address the target set and range of environments of interest. We proved the initial hypothesis to be correct.

This year we focused on four key technical objectives that we believed were important to effectively conclude the project:

(1) To finalize the envisioned modeling and simulations toolkit;

(2) To perform a preliminary system level analysis, and identify security issues associated with standalone unattended ground sensor networks;

(3) To study the possible application of electromagnetic (EM) sensors to the classification problem;

(4) To incorporate the lessons learned to date into our ad hoc network.

## Accomplishments

(1) Finalize the envisioned modeling and simulations toolkit.

This task specifically focused on developing and evaluating algorithms to track multiple targets through a road network utilizing ad hoc sensor networks. We investigated two different tracking algorithms: a graph based algorithm and a particle based algorithm. The performance of graph based algorithms was strongly enhanced when using target classification features as target density increased. The Greedy approach provided a significant computational advantage with only a slight decrease in performance. The particle based algorithm utilized a particle filter approach but required significant changes to the traditional particle filter to meet the objective of tracking targets between sensors. New research included the incorporation of subparticle filter generation at intersections and studies of update interval and sensor spacing requirements for tracking performance. Other advancements to this toolkit included providing an interface to enable the execution of sensitivity studies on high end cluster computing.

(2) Perform a preliminary system level analysis and identify security issues associated with stand-alone unattended ground sensor networks.

We analyzed the network systems' vulnerabilities at the level of common or typical characteristics. Toward this end, we identified three principal attacks a typical adversary might make against a typical system:

(a) Find: Physically touch and recover components of the system for removal, destruction, or possible further analysis and reverse engineering.

(b) Spoof: Acquire the ability to insert false information into the network through the sensors, through communications links, or software control of one or more individual nodes.

(c) Deny: Cause the system to lose some or all of its function temporarily or permanently, typically without finding all the individual nodes, by methods such as jamming communications or neutralizing sensors.

We formed four teams to focus on the security implications of four characteristics of a typical TALON system. These were unattended ground sensor system architecture, network architecture, radio frequency communications, and physical security.

(3) Study the possible application of EM sensors to the classification problem.

Accurate detection of vehicles within a geographic area was a major goal. As such, we sought to analyze and prove that vehicle electromagnetic emissions could be detected reliably across numerous vehicle types and classes. We created a sensor design and developed a new antenna for use with this project. We demonstrated reliable detections of vehicles at a wide range of dis-

tances.

(4) Incorporate the lessons learned to date into our ad hoc network.

The network team successfully upgraded the physical layer (PHY), or radio layer, within the existing network platform. This upgrade allows the network to perform at a higher data rate, 115 kbps, and incorporates a variable transmit power capability. The intent of this task was to prove that technological upgrades to the network could be made fairly seamlessly, with low financial overhead. The network team was able to demonstrate this potential. Moreover, this upgrade was critical for enabling the Virtual Perimeter Security Grand Challenge to forward large amounts of data and images via the TALON network as its foundation. Copyright assertions have been filed for the network codes. Upgraded TALON network hardware is complete.

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# Near Real-Time Characterization for Assured HDBT Defeat

*55079*

*D. R. Gardner, S. Hostetler, B. P. Engler, L. C. Bartel, P. Yarrington, T. W. Pfeifle, L. S. Costin, E. P. Chael, G. J. Elbring, N. P. Symons, K. T. Stalker, D. F. Aldridge, R. E. Abbott, R. T. Gilchrist, R. J. Fogler, A. Mittas, J. M. Opalka, M. T. Oswald, D. A. Dederman, M. E. Bukaty, E. A. Henry, C. O. Landron, R. A. Bates, B. M. Hautzenroeder, R. Hutchinson, J. R. van Houten, M. D. Sena, T. L. Martinez, K. J. Penn Jr., J. E. Lucero, B. L. Roberts, C. B. Webb, J. E. Crepeau, L. Diehl, M. D. Ladd, R. J. Franco, C. B. Luchini*

## Abstract

The project goal was to research the key technologies for a sensor system that would help assure the success of a nuclear earth penetrating weapon strike on a hard and deeply buried target (HDBT) site through rapid geologic site characterization. The approach was to collect penetrability and seismic data using a network of aerially deployed, earth penetrating sensors and active sources and to combine the penetrability data and seismic velocity map. Addressing the technical challenges required a multidisciplinary systems approach and employed Sandia expertise in seismic imaging, air-deployed sensors and sensor networks, earth penetrators, and modeling and simulation.

We developed semi-automated, surface-wave tomography algorithms for sparse, irregular seismic arrays. We determined resolution as a function of the number of source/receiver pairs. We tested the algorithms on field data from a flat, layered site and from a site with significantly varying topography and geology. We investigated methods for detecting small scale scatterers that can indicate subsurface geologic complexity.

We investigated methods for correlating penetrability with seismic velocity to show the near-surface, spatial distribution of geologic material properties. This is a difficult problem because the seismic velocity reflects elastic material properties (e.g., the shear modulus) while penetration is governed by non-linear elastic-plastic properties such as yield strength.

We studied communications architectures and field-tested the formation of ad hoc, high-bandwidth networks for seismic data collection.

We designed a sensor dart and evaluated tools for designing terrabraked flight vehicles. We tested the dart in helicopter drop tests at Tonopah Test Range in harder and softer targets. In the final series we tested functional prototypes that collected seismic data and determined their relative location, compass orientation, and tilt.

*Photograph of the separable penetration probe partially excavated at Antelope Tuff #2. All the electronics survived, and the unit functioned as designed for this target type, demonstrating direct transmission through the soil in real time.*

We designed a penetration probe and drop-tested it at Tonopah in harder targets. The prototype tested direct transmission of deceleration data during penetration into the ground and post-impact data retransmission to airborne and ground receive stations. We conducted static tests of direct transmission to depths typical of softer targets.

## Accomplishments

We reached major technical milestones in the areas of seismic mapping, communications architectures, the penetration probe, and the sensor dart.

In the area of seismic mapping, we developed a semi-automated inversion procedure for surface-wave tomography. We collected field data for a site with significant variations in topography and geology and demonstrated the functionality of the surface-wave tomography algorithms on this data. We showed that moderate degrees of topography have minimal effect

on the seismic tomograms. We also investigated the utility of reverse time migration (RTM) for detecting features below the limit of resolution of surface-wave tomography. And we investigated cokriging for combining penetrability data with seismic data.

In the area of communications architectures, we completed an evaluation of architectures for efficiently exfiltrating large quantities of data and identified an optimal architecture. The optimal architecture uses dynamically formed networks with a posteriori designation of relay nodes, and is scalable. We demonstrated the self-formation of radio networks in the presence of radio interference and with adequate bandwidth performance. We demonstrated global positioning system convergence to within our design objectives in dense foliage (75% sky obscuration) using a Sandia developed algorithm.

The penetration probe is designed to separate into two sections on impact. The forebody must penetrate as deeply as possible given the impact conditions and then record and transmit acceleration data to the aftbody, which is designed to lodge near the surface. We improved the design of the prototype separable penetration probe. In helicopter drop tests at Tonopah Test Range, we demonstrated the aerodynamic stability of the probe and demonstrated that its terradynamic performance met the design criteria: it separated on impact as designed, with the forebody penetrating into the ground and the aftbody remaining near the surface. We demonstrated that the jointed-nose concept works; the concept allows various nose designs to be installed on a penetrator to adapt for varying targets, and also makes manufacturing and assembling the probe easier and less expensive. We demonstrated that the acceleration levels in fore- and aftbodies met the design criteria.

We designed and built two ultra high frequency (UHF) telemetry units and corresponding reradiation units for the penetration probe. These were tested statically and met the criteria for transmission depth. We demonstrated the survivability of the UHF telemetry units and reradiation units in drop tests at Tonopah. We demonstrated intra-probe data transmission in drop tests into harder targets. More specifically, we demonstrated transmission of deceleration data during a penetration event to a receive station at altitude, and we demonstrated retransmission of deceleration data after penetration events to a receive station at altitude and to a ground station.

The sensor dart is intended to embed at the surface of the ground in a near vertical position while protecting its electronics payload. Following emplacement, it must be able to determine its relative location and its orientation (northing and tilt), to collect seismic signals, and to communicate with a relay node via a radio. We designed and built six functional, hand-emplaced test-bed recording units for developing software and for seismic data collection. We improved the aerodynamic and terradynamic design of the sensor dart and designed and built a functional payload. In drop tests of five darts at Tonopah, we demonstrated that we had resolved the significant aerodynamic stability issue for the sensor dart identified last year, and we demonstrated its aerodynamic stability. We demonstrated the terradynamic performance of the surface sensor dart in both softer and harder targets exceeding the design range. We demonstrated the jointed-nose penetrator concept in the sensor dart and demonstrated that the acceleration levels met the design criteria. We demonstrated the survivability and functionality of the air-dropped darts. We verified the functionality of the darts in determining their relative locations, their orientations, their capability to record seismic data, and their capability to communicate with another network radio.

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# Prompt Global Response

*55086*

*R. D. Tucker, A. C. Watts, J. M. Macha, M. A. McWherter-Payne, J. B. Christensen, J. A. Emerson, D. W. Kuntz, R. E. Loehman, J. R. Phelan, R. E. Caicedo, J. E. White, D. D. Stokebrand, N. B. Crane, B. P. Danowsky, B. F. Blackwell, J. Smith, R. B. Bond, S. D. Daniel, J. D. Madsen, A. Ayala, E. E. Creel, J. A. Galloway, M. E. Beader, D. M. Vigil, H. L. Cooper, J. B. Wronosky*

## Abstract

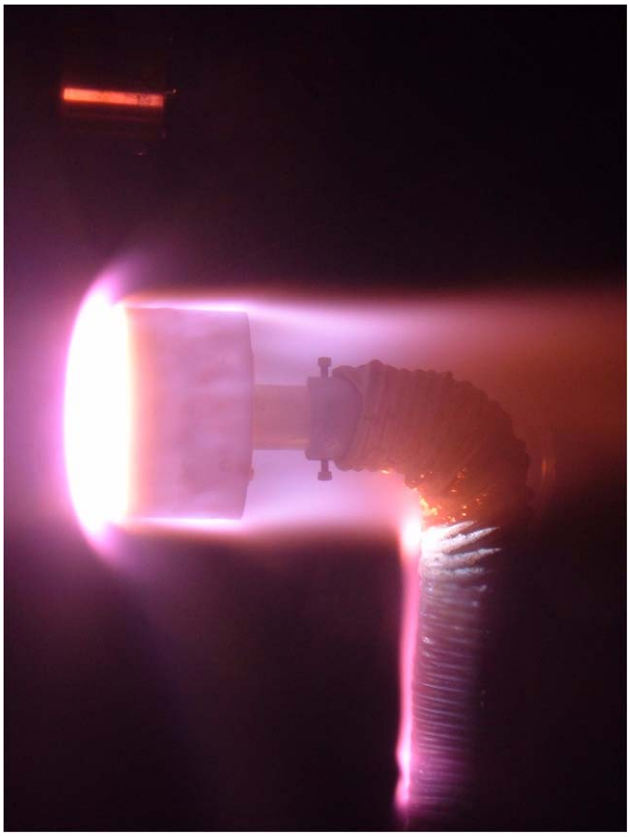
This project addressed the deficiency in certain technologies that are critical to developing a high-speed, long-range, high-precision, autonomous flight vehicle. In generalized quantitative terms, the vehicle must travel thousands of kilometers in tens of minutes with navigational accuracy approaching one meter. The Prompt Global Response (PGR) project was partitioned into three phases, with each phase approximately one year in duration. The initial phase consisted of system studies to define the operational and technical requirements for representative prompt strike missions, followed by assessments of the state of the art of the requisite technologies relative to a desirable level of overall system performance. The second year of the project produced the preliminary engineering design of a “moderate technology stretch” boost-glide vehicle. In this third and final year, the project focused on applied research in the key boost-glide technology areas of navigation, guidance and control, and vehicle thermal protection. In addition, we carried out a complementary activity of new computational model development to support these two major thrust areas.

## Accomplishments

- Developments in Navigation, Guidance, and Control

*Advanced Control System:* We applied dynamic inversion (DI) methodology to the design of flight control algorithms for a high-beta hypersonic vehicle with three axes control. DI provided a more structured approach and yielded a control design with superior performance compared to designs based on classical approaches. We delineated difficulties in applying the DI method to a vehicle with only two axes control and assessed candidate approaches.

*Carrier-Phase Global Positioning System (GPS) Accuracy Enhancement:* We developed a new GPS carrier phase solution algorithm capable of achieving sub-meter navigational accuracy over long distances.



*Arc jet test of carbon-carbon oxidation protection material developed in the Prompt Globlal Response thermal protection task.*

Application of this new algorithm required development of new GPS receiver software and complementary Kalman filter processing algorithms. Prototype versions of this system provided sub-meter level accuracy during laboratory radio frequency simulations and ground testing.

*Precise Ephemeris GPS Accuracy Enhancement:* This task explored the feasibility of eliminating GPS satellite position and clock errors by making precise, real time ephemeris data available to the on-board vehicle flight computer. A system conceptual design

included the precise ephemeris data message content, link margin analysis, on-board receiver design, and receiver development schedule.

*New Commercial Off-the-Shelf (COTS) Architecture Flight Computer:* The IMPACCT II computer, based on COTS architecture, was developed as a prototype system to meet hypersonic vehicle needs over the next decade. The prototype successfully completed functional testing and thermal characterization. We tested the computer for susceptibility to proton single event upset. The results fill an industry gap in understanding radiation effects on COTS based computer design.

- Developments in Vehicle Thermal Protection

*Organic Based Glass Precursors for Oxidation Protection:* We developed materials and processing methods for providing oxidation protection for carbon-carbon (C-C) composites. We tested protection schemes using siloxane based infiltrants and coatings in bench scale ovens and at the National Solar Thermal Test Facility solar tower. These materials decomposed to form silicon based glasses. The best performing protection scheme tested at 150 W/cm$^2$ for 10 minutes reduced the mass loss from a carbon-carbon sample from 70% to 20%.

*Oxidation Protection Using Ceramics:* This task focused on developing external ultra high-temperature ceramic coatings. We used liquid precursors to deposit diboride films on graphite and C-C composites. We tested coated specimens in a 1700 °C furnace at the National Solar Tower Test Facility. Solution deposition techniques gave promising results and are excellent candidates for further development. The best candidate coatings incorporate Zr or Hf diborides, a silica former, and a borate former.

*Solar Thermal Capability for Material Screening Tests:* We established a new capability for testing thermal protection materials at the National Solar Thermal Test Facility. We designed and fabricated fixtures to test samples at heat fluxes of up to 200 W/cm$^2$ for 10 minutes. Interchangeable mounting panels provide the flexibility to test individual samples or up to 12 samples simultaneously. This facility can match the flight environments experienced by hypersonic flight vehicles better than bench scale equipment and at a lower cost than arc-jet testing.

*Developments in Modeling and Simulation:* Accurate prediction of the aerothermal environment for hypersonic vehicles is essential for establishing design criteria for thermal protection systems. We extended Premo, a computational fluid dynamics code that formerly used an ideal gas assumption, to include equilibrium chemistry modeling. We also developed a new model in the Chaleur heat transfer code for the thermal and pressure response of a charring ablator subjected to an aerodynamic heating environment. We implemented the energy equation using a residual formulation and verified it on a moving/deforming grid. We also computationally investigated key parameters that affect the range for hypersonic, maneuvering reentry vehicles. We simulated the effects of trajectory tailoring, velocity at reentry, vehicle weight, lift-to-drag ratio, and booster hand-off altitude. We developed a method for including aeroheating information in preliminary trajectory design and optimization. An example application showed that it is feasible to include aerothermal constraints to reduce design cycle iterations.

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# Winning the War: A Systems Approach to Defending Our Borders

*55087*

*C. A. Pura, L. Shyr, H. R. Ammerlahn, M. J. McDonald, R. P. Tsang, R. G. Hillaire, D. K. Belasich, P. K. Barr, R. R. Baxter, M. A. Ehlen, P. B. Merkle, A. Vaughn*

## Abstract

We developed a suite of simulation tools that make strong contributions relevant to the analysis capabilities of the Department of Homeland Security (DHS). The simulation tools provide a systems view of defense architectures and allow a user to rapidly design, evaluate, and optimize these complex systems. Specifically, these tools could be of significant value in evaluating and optimizing threat detection and response systems used at US borders and aviation facilities (for example, simulations for wide area unconventional nuclear threat detection systems and chemical and biological crises management systems). These simulation tools provide analysts with a valuable, high-level understanding of system performance and the way in which individual components (e.g., technology and policy) of a system can work together most effectively. Furthermore, the hierarchical structure of the simulation will allow analysts to evaluate the contributions and multiple interactions of each component in the system.

## Accomplishments

We developed working models of an airport, a pedestrian crossing, a vehicle crossing, a truck cargo, and a seaport container operation. We added the functionality to federate discrete event and continuous time models through a high level architecture interface in order to perform mixed fidelity analysis. We exercised a mixed fidelity model based on a trade lane analysis to test and demonstrate the model's capability to analyze stock and flow, and sensor placement effectiveness. We also used the sea trade lane as a basis to investigate the use of economic modeling, policy, and technological impacts of the Container Security Initiative. We found that the impact of delays in shipments through the Port of Tacoma that exceed (on average) 6% of the travel time will cause a change in customer/supplier behavior and have a material effect on the gross domestic product. DHS organizations have indicated an interest in providing funding to continue this work.

# Integrated Fiber Lasers for Efficient High Power Generation

*81752*

*D. A. Kliner, R. L. Schmitt, G. A. Vawter, A. V. Smith, W. J. Thomes Jr., W. R. Bolton Jr., G. R. Hadley, R. L. Farrow, J. P. Koplow, J. K. Kim, A. A. Hoops, B. T. Do, P. E. Schrader*

## Abstract

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, this 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, and numerical modeling) to enable miniature, ultra-efficient, high power devices that will revolutionize the application of lasers to real world problems.

This GC research project comprises five primary technical areas:

(1) Rare earth-doped fibers and fiber amplifiers (including pumping methods and output-beam formatting);

(2) pump diodes;

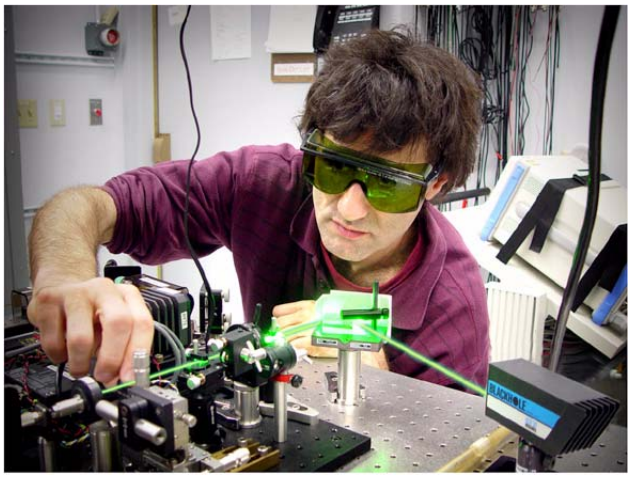
(3) microlaser seed sources;

(4) waveguide and laser/amplifier modeling; and

(5) system engineering and testing.

## Accomplishments

We achieved accomplishments in each of the five primary technical areas that encompass the key components of a high power, fiber based laser system.



*Sandia researcher Dahv Kliner and his team developed a bend-loss-induced mode filtering method that allows the use of large core diameter fibers while maintaining high beam quality.*

- Rare earth doped fibers and fiber amplifiers

The primary goal is to demonstrate significant power scaling by extending the mode-filtering (coiling) technique. Specifically, we proposed to reach a peak output power of 1 MW from a single fiber with diffraction-limited beam quality; we also proposed to achieve stable, linear output polarization.

We obtained a peak power of > 1.2 MW (0.57 mJ pulse energy in a pulse duration of ~0.45 ns), thereby exceeding our target. The initial experiments had a beam quality that was twice the diffraction limit (M2 = 2.1) because of a defect in the fiber. Subsequent experiments with a new fiber achieved diffraction-limited beam quality (M2 < 1.2) with even higher pulse energy (> 1 mJ). We recently obtained a fiber with a similar design that includes stress rods to provide polarization-maintaining operation.

- Pump diodes

The goal of the laser diode (LD) effort is to develop devices with substantially better spectral and thermal characteristics than commercially available LDs. The primary task was to investigate potential laser designs via evaluation of current technologies and modeling. The team proposed several design concepts

and selected three candidate designs to fabricate: an alpha-DBR (distributed Bragg reflector) laser, a monolithically integrated master oscillator power amplifier, and a grating-stabilized tapered laser. These novel LD designs show a great deal of synergy because the processing platforms can be shared among the various device implementations.

- Microlaser seed sources

The goal of the seed source development effort is to design and build miniature, high performance pulsed lasers to seed fiber amplifiers. The primary objective was to build a Nd:YAG microlaser (1064 nm) with 0.1 mJ pulse energy, ~2 ns pulse duration, greater than 1 kHz repetition rate, diffraction-limited beam quality, and single longitudinal-mode operation. We built a laser that met or nearly met all of these criteria. We used this laser to seed a mode filtered fiber amplifier and provided our highest pulse energy to date. We also began design work on 1030 and 1535 nm microlasers.

- Waveguide and laser/amplifier modeling

We developed models that calculate the transverse mode profiles of bent fibers. We learned that when a step-index fiber is coiled tightly enough to filter out high order modes, the fundamental mode is substantially distorted and has a smaller effective modal area. This result has profound implications for the optimum fiber design and for the influence of nonlinear processes.

Our initial calculations indicated the importance of treating gain and loss in a bent fiber in an integrated and rigorous framework. We thus developed vectorial and semivectorial beam propagation codes that can calculate the evolution of the optical field through several centimeters of fiber. These new codes represent state-of-the-art capabilities that are not available in any commercial package. Specifically, the combination of first principles bend-loss analysis, modal gain resulting from arbitrary refractive index and rare earth dopant profiles, and integrated treatment of nonlinear optical effects is unprecedented in the fiber laser/amplifier field. This approach was not anticipated, but arose from our recognition of the need to treat these phenomena in a consistent framework.

- System engineering and testing

The overall goal of the engineering and testing effort is to establish the basis for transition of technologies to applications. We performed vibration tests of a side-pumping module. We also performed radiation tests of a suite of composition-characterized Yb and Er doped fibers.

## Refereed

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# Advanced Fusion Concepts: Neutrons for Testing and Energy

*81753*

*T. A. Mehlhorn, G. E. Rochau, M. A. Ulrickson, R. A. Causey, J. S. Arzigian, R. M. Huelskamp, A. B. Baker, T. E. Drennen, T. L. Sanders, P. J. Griffin, G. A. Rochau, J. R. Lee, C. J. Garasi, T. R. Lockner, 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, W. C. Hines, C. L. Ruiz, J. E. Bailey, D. L. Hanson, M. E. Cuneo, D. B. Sinars, M. P. Desjarlais, T. A. Haill, D. R. Welch, B. V. Oliver, M. A. Christon, A. C. Robinson, R. A. Vesey, R. B. Campbell*

## Abstract

This project is evaluating novel, advanced fusion concepts that may quickly lead to sufficient neutron outputs useful for effects testing and energy production purposes. Achieving equivalent outputs using the indirect drive inertial confinement fusion (ICF) approach pursued over the past several years does not appear to be on the near horizon, despite significant progress. The project has four tasks: (1) theory, modeling and simulation; (2) experimentation; (3) neutron applications; and (4) proliferation implications. The theory, modeling and simulation task is developing and applying radiation-MHD (Magnetohydrodynamic) and hybrid fluid-PIC (particlein-cell) plasma physics models to evaluate the underlying physics and performance of these novel fusion concepts.

The experimentation task is focused on developing (1) advanced diagnostic techniques to measure the plasma conditions at fusion time, and (2) cryogenic systems to enhance performance. The neutron applications task is exploring the neutronic feasibility of a neutron multiplier assembly coupled to a Z-pinch neutron source, i.e., a Z-pinch driven nuclear assembly (Z-EDNA), with the goal of achieving a radiation testing environment equivalent to the Sandia pulsed reactor. Finally, the proliferation task is focused on evaluating the proliferation considerations associated with these novel concepts, including implications for existing non-proliferation regimes.

*Researcher Daniel Sinars demonstrates the setup he and his team created to peer into the center of Sandia's Z machine at the moment of firing. The crystal under his finger is attached to portions of a Z target configuration.*

## Accomplishments

The key scientific goal of this project is to evaluate the viability of advanced fusion concepts that could produce sufficient neutrons for testing and energy applications on a timescale that is shorter than that for the present hot spot ignition targets in the baseline ICF program. The project team organized around four major tasks that addressed the following key technical issues:

(1) The neutron yield from our initial advanced fusion target experiments is roughly 1–2% of two-dimensional (2-D) radiation-MHD code predictions. What are the physical mechanisms that are causing this degradation? Does viscously-damped MHD turbulence directly heat the fuel ions and alter both the fusion reaction rate and the pressure balance of the core?

(2) The physical degradation of the neutron yield is likely to be occurring at the stagnation of the imploding fuel. Can we develop gated imaging and spectrally-resolving detectors that provide data on the fuel conditions at this time? Cryogenic fuel is predicted to give higher neutron yields. Can we develop a liquid deuterium cryogenic capability for fusion experiments on Z?

(3) Can the stockpile-to-target sequence requirements for neutron testing be met by a Z-pinch source multiplied by a low enriched uranium subcritical blanket within the physical structure of Z? Can a Z-externally driven nuclear assembly system replace the Sandia pulsed reactor for defense programs testing?

(4) What are the proliferation concerns related to advanced fusion concepts?

We made significant progress in each task area:

- Theory, Modeling and Simulation

We began testing of a 2-/3-D radiation-MHD simulation capability in ALEGRA-HEDP (High Energy Density Physics) for application to the study of neutron yield degradation mechanisms in advanced fusion targets. We initiated a task to add fusion burn and alpha particle transport to ALEGRA in order to directly compare experimental neutron yields. We began the development of a hybrid plasma simulation capability and are applying it to the study of heating mechanisms that were hypothesized to occur in the stagnated plasma due to viscously damped MHD turbulence. We also developed a linear analysis of these MHD instabilities. We determined that fast ignition is not the most promising ignition concept, and it will be dropped from our research to maximize progress on the remaining option.

- Experiments and Diagnostics

We embarked on the development of a number of new diagnostics or diagnostic improvements designed to help us evaluate the final stages of liner implosions. Most of the development effort focused on building high spectral resolution spectrometers using spherically bent crystals and gated detectors. We conducted a large number of hardware cooling tests to help design a cryogenically cooled liner experiment, and also made one unsuccessful attempt at fielding such a test on the Z facility. Based on those tests, we redesigned most of the hardware and plan to attempt cryogenic tests on the Z facility.

- Neutronics (Z-EDNA Concept)

Results from our neutronic scoping studies for the Z-EDNA concept using a 14-MeV, D-T pulsed neutron source show promise for achieving the levels of defense program testing requirements. Conservative neutronic calculations show that the neutron fluence and pulse width requirements could be met by Z-EDNA, driven by a fusion neutron source on ZR that uses a 20% enriched (low-enriched uranium) multiplier assembly operating at a keff between 1.000 and 1.006.

- Proliferation Issues

We compiled and analyzed over 100 source documents of various types regarding worldwide fusion research and the applicability of various arms control treaties to limit such research that could have proliferation concerns. We examined key enabling technologies associated with potential military applications of fusion research. We also examined existing arms control treaties and possible future regimes that could potentially limit, or stop, possible military applications of the technology.

# Microsystems-Enabled Virtual Perimeter Security

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*R. W. Stinnett, N. M. Berry, K. T. Stalker, F. J. Oppel III, D. A. Pritchard, D. G. Adams, H. D. Nguyen*

## Abstract

The purpose of the Virtual Perimeter Security (VPS) project is to demonstrate the value of "beyond the fence" detection and assessment of intruders around facilities. Existing and emerging sensing, data processing, and communications capabilities, including hardware, software and algorithms, make it possible to develop and field such systems affordably.

Guidance from the VPS External Advisory Board (EAB) indicated that such capabilities would be valuable as a way to provide early warning of intrusion. They suggested that the highest value implementation of this capability would be in non-line-of-sight (non-LOS) areas.

The VPS application has several attractive features that are different from many other sensor system applications. The most difficult elements in fielding sensor systems for urban operations and use in hostile territory are covert emplacement, communications, and power. For VPS, the system is fielded on terrain owned by the facility and is at least nominally off limits to the general public. Node emplacement and tuning of communication links can be done by hand. Because the system does not have to be covert, power can be provided by solar arrays and batteries. These differences provide enormous advantages in VPS applications.

However, VPS also has some objectives that are more difficult to satisfy. The system must provide enough information to human operators to enable them to know how to respond. This may be done through multi-modal sensor suites coupled with local data processing, a multi-level data fusion architecture, and communications links that provide filtered information to a remote operator. In addition, VPS systems must be affordable enough to field at the sites and flexible enough to adapt to changing threats. We developed a detailed VPS concept and fielded a demonstration of the value of this capability at a relevant site.

## Accomplishments

We accomplished the following:

(1) We followed EAB guidance to focus on field demonstration of the VPS value in non-LOS areas around facilities.

(2) We developed specifications for system performance and success metrics for the demonstration.

(3) We developed and tested algorithms and systems to detect and distinguish humans and small vehicles with low nuisance alarm rates (NARs).

(4) We focused modeling on system level data fusion and NARs.

(5) We developed and demonstrated a scalable and customizable hardware and software platform that can meet site needs.

In the VPS demonstrations conducted in September 2005, we successfully showed:

- the ability to detect human and vehicle intrusion at non-LOS areas;
- high probability of detecting intruding people;
- high probability of detecting intruding vehicles;
- the ability to distinguish footsteps, dramatically reducing NARs;
- the ability to do visual assessment triggered by physical sensors;
- robust radio frequency communications to non-LOS areas;
- a self-powered operation and tamper detection;
- an integrated system functionality with operator interface.

The system is also designed to accommodate other relevant features to meet site-specific needs.

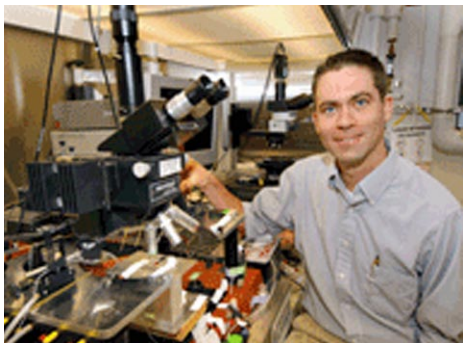
# Corporate Objectives

The Corporate Objectives investment area 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 Objectives 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.

**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 **80838 "Molten Salt Based Growth of Bulk GaN and InN"** demonstrates the feasibility of the bulk production of a novel electro-optical material used in advanced applications. Researchers are working to resolve the problem of the lack of low defect density, large area substrates for epitaxial thin film growth. Current approaches to the growth of GaN require high pressure and high temperature, take significant time to grow, and generally have high defect density. Using a molten salt based solubility growth technique for crystal synthesis, researchers grew 1 mm diameter GaN crystals at atmospheric pressure in one afternoon. Inexpensive bulk production of GaN and InN opens the way to much broader applications of these materials in such diverse areas as solid-state lighting and synthetic aperture radar.

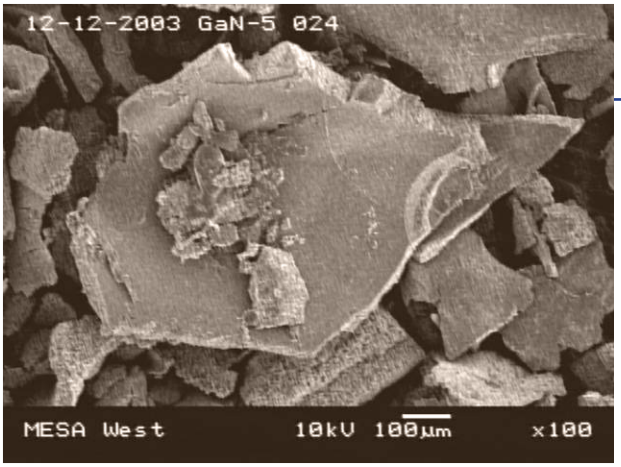
Investments in Corporate Objectives also enable **University Collaborations** to create new science and engineering technologies, to employ complementary resources and skills for achieving common goals, and to recruit and guide top students for Sandia's work force.



**80592 "Ultrafast Low Voltage MEMS Switches for Optics and RF Applications"**

Researcher Gregory Nielson is working to develop a new actuation switching technique for MEMS that can be used to create vertical or horizontal rectilinear motion or torsional motion.

For example, the Truman Fellowship Award supported principal investigator Gregory Nielson on project **80592 "Ultrafast Low Voltage MEMS Switches for Optics and RF Applications."** Researchers on the project are developing a completely novel actuation technique for a microelectromechanical system (MEMS) switch structure that will provide switching times of 100 ns, require actuation voltages of less than 10 V, and provide a switch displacement of at least 1 $\mu$m. This innovative technique enables significant performance improvement over any other MEMS actuation method. Researchers plan to demonstrate both radio frequency (RF) and optical switch prototypes. These high speed switches will facilitate small, power efficient, high-speed switching networks. Applications include nuclear stockpile applications, remote sensing, satellite operations, and power efficient communications networks.




**80838 "Molten Salt Based Growth of GaN and InN"**

Scanning electron microscopy image of 1 mm diameter GaN crystal grown at atmospheric pressure in one afternoon.

# Advanced Concepts

## The Observer

*79737*

*M. G. Stickland, H. E. Link, R. L. Craft, G. M. Pollock, L. D. Cumiford, N. D. Pattengale*

### Abstract

Since the events of 9/11, a significant amount of attention has been paid to increasing our intelligence community's (IC's) ability to "connect the dots." This is a multifaceted problem involving the IC's collection regimes, processing methods, analysis approaches, and dissemination mechanisms. The intelligence analysis aspect of this problem is interesting because of its heavy reliance on human performance to succeed and the attendant vulnerability to personal biases, inexperience, or finite capacity of humans who perform this work. A central task in this problem space is determining which indicators of activity would best confirm or refute hypotheses that existing evidence would seem to suggest. This report describes our efforts to research and demonstrate the feasibility of using a software-based tool to assist analysts in this process, and to strengthen existing and proposed data mining technologies by providing higher quality operational signatures.

To achieve these goals, we first explored the integration of artificial intelligence (AI) plan generation software with agent-based hybrid dynamic system (ABHDS) simulations. In this context, a scenario produced by AI planning software functioned as a script that the simulated actors had to play out using simulated behavior modes. We then described a technique developed for detecting robust and persistent behavioral signatures indicative of the simulated scenarios in the ABHDS simulations. The behavioral signatures could then be applied towards identifying the existence of the scenarios within IC data collections using data mining techniques.

### Accomplishments

We began with the hypothesis that certain existing signature detection techniques could be improved through automation. The techniques in question rely on simulations modeled as ABHDSs. A bottleneck in this process is the manual coding of these models. We planned to automatically generate ABHDS models using discrete scenarios produced using AI planning software and a library of reusable continuous-actor-mode (CAM) simulation model components. To prove our hypothesis, we set out to demonstrate that implementation of the capability is feasible.

We utilized the SIPE-2 AI planning software from SRI International to generate the discrete scenarios that would be simulated and analyzed to detect their operational signatures. We also utilized signature detection techniques developed by Rich Colbaugh (New Mexico Institute of Mining and Technology) and Kristin Glass (National Center for Genome Resources). Colbaugh and Glass collaborated with us to help develop automated capability and to adapt their techniques to the highly dynamic domain of terrorism.

Our design comprises a component-based architecture that takes the planner's output as input to a component we call the SCONE (Simulation CONfiguration and Execution). The SCONE first converts the plan into a data representation of the discrete supervisory control portion of our target ABHDS model. Secondly, the SCONE maps the behavior modes contained in each plan action node to pre-built data representations of their corresponding CAM components. Finally, the SCONE constructs a complete model for execution by a simulation execution system (SIM). This approach requires a completely data-driven SIM for running our automatically generated ABHDS models.

After developing our requirements for the needed SIM, we conducted an extensive survey of available ABHDS capable simulation software packages. After reviewing candidate systems for desired functionality, we selected AnyLogic from XJ Technologies as the only system meeting requirements for further testing and evaluation. Our next goal was to flesh out the requirements of the SCONE component, given a SIPE-2 planning object as input and an AnyLogic ABHDS simulation model as our target output. To accomplish this goal, we first developed a test case Red Team scenario of a hypothetical terrorist operation. We then translated the scenario into a plan format (a partially-ordered directed graph representation of the scenario similar to what SIPE-2 would produce), and determined which simulated behavioral modes to assign to each plan action node. The behavior mode designations provide the connection points the SCONE uses for mapping plan action nodes to pre-built CAM components when dynamically constructing a simulation model.

Since AnyLogic is able to import the discrete supervisory control portion of ABHDS simulations from Microsoft (MS) Project plan information exported to MS Access files, we determined that this technique would be a viable first proof-of-concept of our SCONE component. We developed an MS Project representation of our test case scenario, including the behavior mode designations within each plan action node, and simple CAM models in AnyLogic for each behavior mode in our test. We then imported the plan into AnyLogic, mapped the CAM components to their respective elements in the imported supervisory control model, and executed it to simulate our scenario.

Through this process we confirmed our original hypothesis that certain existing signature detection techniques can be improved through automation. In addition, we determined that the following goals are feasible: (1) developing a means of automatically generating ABHDS simulation models from AI planning generated discrete scenarios, and (2) developing a library of reusable CAM components, is feasible. We feel this approach is indeed useful for improving existing signature detection techniques.

Further, we developed a SIPE-2 planning domain theory and successfully generated a plan equivalent to our manually developed test case. In doing so, we created the Capability-Resource Decomposition modeling approach, a nascent modeling technique designed to facilitate rapid formation of certain aspects of terrorist operation scenarios as domain theories. In parallel, we developed and tested more complex CAM models and used them to help adapt the existing signature detection techniques to the domain.

# Adaptive Software for Advanced Human/Computer Systems

*79738*

*A. M. Bouchard, C. M. Johnson, G. C. Osbourn*

## Abstract

Consider the following problems: (1) An analyst wants to customize certain analyses provided by her system, but either the system does not support customization, or it is too hard to understand how to make the system do what she wants. (2) She also wants to analyze data from a new database. Her existing analysis tools won't talk to the new database. (3) 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. All of these and a host of other problems have a single root cause: the software only knows how to talk to, process, and present information in the single way it was programmed. Any modification of the software must be made by programmers. The cost and time scale for changes directly impact user productivity. In this project, we are developing a user-adaptable computing environment in which the user can modify the environment him/herself on-the-fly: specifically, the data that are accessed, how that data are organized, and the algorithms (programs) that can be run on that data. Essentially, the user acts as the programmer without having to know the underlying programming language. The user specifies a task using powerful English-like verbs, and the code to carry out the task is self-assembled under-the-hood using our new self-assembling software technology. In the first year, we began implementing a proof-of-concept programming capability along with innovative user interface and data organization capabilities. In the next two years, we will focus on a specific national security application, although the toolkit of capabilities that will be developed simultaneously will be generally applicable to any software application. We intend to revolutionize the way software interacts with people, leading to vastly improved performance of human/software systems.

## Accomplishments

The first accomplishment was to identify the scope of the proof-of-concept system to be developed in the first year. It will provide to the user: (1) the power of programming without the language, syntax, and minutiae; (2) an intuitive, memorable, adaptable way of organizing data; (3) the capability to find, share, change, reuse, and undo anything, anywhere; and (4) the ability to specify actions launched automatically in response to a specific situation any time it occurs in the future. Specifically, we limited the scope by limiting the programming capability to simple calculations and the analysis of tabular data.

Next, we completed designs of the user interface, the underlying self-assembling software (SAS) building blocks, and the code-generation capability that translates the user's specifications into specific self-assembled code. This was a very large (many man-month) task and a really substantial accomplishment. A technical advance was also submitted.

Implementation of the user interface and code-generation is under way. At this point, the code-generator can generate some simple "find" analyses; but in order to do this, substantial code-generator infrastructure had to be implemented. Thus, a significant fraction of the code-generator code is complete. Implementation of the SAS building blocks needed for the first iteration is also complete. Code to render objects on the user interface has also been implemented. We are well on our way to the completed first iteration of the adaptable computing environment.

# System Dynamics Modeling of International Border Permeability and Gradient Dynamics

*79739*

*H. D. Passell, V. C. Tidwell, L. A. Malczynski, C. M. Johnson, J. G. Turnley, L. A. McNamara, N. A. Bowles, S. H. Conrad, G. C. Newman, P. H. Kobos, J. R. Brainard, M. A. Ehlen, J. N. Emery*

## Abstract

Political borders are controversial and contested spaces. As an attempt to better understand movement along and through political borders, this project applied the metaphor of a membrane to look at how people, ideas, and things "move" through a border. More specifically, the research team employed this metaphor in a system dynamics framework to construct a computer model to assess legal and illegal migration on the US-Mexico border. Employing a metaphor can be helpful, as it was in this project, to gain different perspectives on a complex system. In addition to the metaphor, the multi-disciplinary team utilized an array of methods to gather data including traditional literature searches, an experts workshop, a focus group, interviews, and culling expertise from the individuals on the research team. Results from the qualitative efforts revealed strong social as well as economic drivers that motivate individuals to cross the border legally. Based on the information gathered, the team concluded that legal migration is not a dynamic system and hence, available demographic models sufficiently capture that story. Results from both the quantitative and qualitative data searches were used to modify a 1977 border model to demonstrate the dynamic nature of illegal migration. Model runs reveal that US policies based on neo-classic economic theory have proven ineffective in curbing illegal migration, and that proposed enforcement policies are also likely to be ineffective. The model results reflect items in the current literature that suggest demographic and economic changes within Mexico are likely to slow illegal migration by 2060, and that waiting may be the most efficient policy.

## Accomplishments

The project was successful in showing that system dynamics modeling approaches can be used for modeling dynamics associated with international borders in general, and with the US-Mexico border in particular. The project used the structure of a border dynamics model built by Dabiri in 1977 and modified that model to include new understanding of border dynamics and to include new data. Prior to the modeling effort, the project gathered experts from around the US-Mexico border area and held a meeting as a way of consolidating information and perspectives. Members of the project team also traveled to the border region and held focus group meetings on the US side of the border and interviews with US citizens and officials in the border region. The modeling effort showed that the dynamics of multi-disciplinary subjects, like the border, that include economic, social, physical, and cultural systems, can be better understood through system dynamics modeling. The model was used to test various policy options with regard to border management.

# Understanding Human Interactions with Complex Adaptive Systems

*79740*

*S. Tucker, B. E. Hart, M. J. McDonald, E. P. Parker, F. J. Oppel III, C. E. Lippitt, T. Smith, P. G. Xavier*

## Abstract

Complex Adaptive Systems (CASs) that include people, networked sensors, and response agents, such as mobile weapon platforms, are increasingly envisioned as approaches to dealing with today's national security issues. While people can have significant impact on system behavior, with more complex systems, we lack the understanding needed to formally address the human contribution to these complex systems. Without comprehensive system understanding including human behaviors, CAS approaches will be unsuccessful in addressing evolving threats. Complex system-level behaviors (i.e., emergence) that result from networking multiple heterogeneous adaptive assets make purposeful human involvement difficult.

To build a core understanding of the behavioral attributes of human-CAS interactions, we conducted experiment-based exploration using existing virtual environments (Umbra), "Evolutionary Experimental Engineering" (tight spiral design and analysis) of these systems in short simulation studies, and immediate feedback from subject matter experts (SMEs). These experiments explored complex system-level behaviors in overall CAS performance, including: (1) command and control by the protective force and (2) the impact of critical design and operational parameters on specific measures of effectiveness that were provided by the Department of Energy (DOE) physical security community. We performed approximately 15 of these design/analysis spirals during a 5-month period that led up to a large integration "Summer Study."

The Summer Study effort integrated multiple simulation technologies from many different projects into a more comprehensive simulation environment. Additionally, a government off-the-shelf (GOTS) user interface, SOFViz, was integrated onto the Umbra physics engine allowing SMEs direct interaction with the simulation environment, e.g., through drag-and-drop interfaces with live realistic physics effects. A simulation study with the New England Complex Systems Institute (NECSI) showed that by using new network analysis algorithms, ad hoc networks with adaptive radio frequency (RF) power and topologies could achieve significant power/battery savings over many current implementations. This is significant because battery usage is a major problem in the military today. This work continues to take lessons from social and biological modeling and apply them to real technical problems.

## Accomplishments

The process of Evolutionary Experimental Engineering consists of multiple spiral development efforts to produce an integrated simulation of all the components. The initial simulated scenario had a team of ten men with vehicles attacking a facility. The guiding metric was the accuracy speed of threat detection. Each avatar, representing a human in the simulation, was equipped with a visual sensor that acts as the eyes and allows the team members to "see" or detect other team members, emergency response vehicles, and nuisances; this enables appropriate response behaviors. Sensing included four tracking radars, one placed at each corner of the facility. Virtual Perimeter Security (VPS) cluster circles of six nodes were placed in locations without line of sight to the facility. Each node of a VPS cluster had one magnetic and one seismic sensor and three imagers to provide a 360 degree field of view (FOV).

The initial spirals started with the SMEs developing an adversary attack plan that included their routes and timing. However, the plan was scripted based on perception, and the avatars could vary from the script. Following spirals added increased sensors (Radar, VPS, and additional VPS). Within these spirals were sub-spirals with the insertion of false alarms, nuisances, and data fusion algorithms. The final simulation contained fourteen VPS clusters, four tracking radars, two relays, one communication tower, a seven man assault team, three snipers, two emergency response vehicles, and eight horses as nuisances.

This project allowed us to develop the realistic representations of adversaries from SME discussions. Secondly, it increased our understanding of sensors and provided information on the importance of the data fusion algorithm and early detection of threats. Thirdly, integration of all of the technologies allowed us to study the interactions of these technologies with human behaviors. The initial work in this project led to a

larger, cross project integration activity we called the Summer Study, which was an effort to bring together technologies from several different projects that were based on the Umbra framework. The work that was done on this project provided a scenario "seed" that was enhanced by the inclusion of technologies from other projects.

The first step was to enhance the character models used by Sandia's Advanced Concepts Group (ACG) with actual behaviors. These behavior-driven characters were then used in scenarios that included sensors for detecting them, communications between sensors and a command and control system, and the tasking of responders by the command and control. For response, the Organic Aerial Vehicle (OAV) model was used. It was equipped with a sensor that could detect these characters and thereby give the command and control center eyes-on-target and capability. The Summer Study effort successfully explored ways to integrate technologies from many different projects to produce a more comprehensive simulation of this complex environment.

While the Umbra developers are working to make the tool more user friendly, it is still mainly a tool of the computer scientist. A second track was started to integrate the three-dimensional (3-D) terrain viewer SOFViz with Umbra. SOFViz is very intuitive for the end-user (i.e., the SME). We produced a demonstration of SOFViz in which we can drag and drop sensors, click down paths (adversary, robots, etc.), and have Umbra generate realistic interactions between these entities.

A simulation study with NECSI showed that by using new network analysis algorithms, ad hoc networks with adaptive RF power and topologies could achieve a 90% power/battery savings over many current implementations. This is significant because battery usage is a major problem in the military today. A paper on the ad hoc network study is being prepared for publication.

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# Algorithms for Attention and Recognition of Humans and Threat Objects in Video

*86363*

*P. B. Merkle, G. L. Jones*


## Abstract

The assessment of intrusion detection alarms in secure areas requires human inspection by closed-circuit camera, complemented by security patrol response. Video inspection of the alarm area is typically conducted at a Central Alarm Station (CAS), aided by replay of stored video or use of pan-tilt-zoom (PTZ) cameras in real time. For large secure areas, video assessment can become challenging due to the potential for difficult viewing conditions, multiple alarms, and operator fatigue. We began the development of a novel intrusion detection system based upon machine visual attention and object recognition algorithms. Methods to rapidly capture useful imagery for algorithm training and a video database of weapons types were developed. A model secure interior room and model secure exterior area were used. Assault rifle, submachine, and handgun weapons were digitally recorded for calibration partition into separate algorithm development and validation data sets. A series of simple human and background motion sequences was taken in a model secure perimeter to explore the types of footage needed for algorithm development. These method development sequences were necessary to understand the experimental needs for scenes for background motion and for human motion with weapons. Single images were manually extracted from the indoor testing video sequences to constitute the testing set for object recognition experiments. The object detection rate was 65% for the MP5 submachine gun, 46% for the Uzi, and 15% for the Glock pistol, with about a 0.5% per image average false positive rate. A motion attender/detector (MAD) algorithm was developed using ideas from object recognition, saliency, and “surprise” theory.


## Accomplishments

We developed a video database of adversary and protective force firearms and relevant interior and exterior motion sequences for algorithm development and testing.

We evaluated the performance of an industry state-of-the-art scale invariant feature transform (SIFT) based object recognition algorithm for the detection and recognition of firearms in partially cluttered interior settings. The algorithm detection rate was 65% for the MP5 and 46% for the Uzi when weapons were carried in a variety of postures. The pistol was detected in only 4 of the 26 testing images (a 15% detection rate).

*Video clip of tracking humans across a natural environment.*

Major factors affecting the performance were:

- clutter in the firearm background
- image resolution
- amount of occlusion
- amount of unique local features of the firearm

Future research should focus on ways of removing the dependency on the background and further reducing the false positive rate.

We developed a MAD algorithm using ideas from object recognition, saliency, and “surprise” theory. A motion classifier algorithm, currently in development, attempts to flag human motion when it is present, and is designed to be able to distinguish between different types of human motion. However, the classifier requires, as input, relatively clean, uncluttered video sequences in which only one large object is moving. The goal of the MAD algorithm is to simplify the task of the classifier by effectively removing the background scene of a video from consideration and also by highlighting features in present foreground objects. The MAD runs several simpler subtasks in parallel and then combines their results in order to produce its end

output, a set of rectangular regions on an image plane (for each video frame) together with some features found inside those regions. The subtasks are: (1) detecting pure motion, (2) detecting foreground objects, and (3) detecting “surprising” regions.

The goal is to detect objects that are simultaneously moving, new to the scene, and somehow surprising to look at. Using a metric suitable for the given task, each of the MAD’s three subtasks ultimately produces a saliency map over the original image indicating regions where there are large amounts of conspicuity. The maps are combined linearly into a master saliency map, a single surface over the recorded image plane. Large, spatially connected, and highly salient regions on this master map are outlined with rectangles using a simple clustering algorithm that excludes regions with insufficient saliency mass. The foreground features computed by subtask (2), and lying inside these rectangular outlines, are ultimately stored away for use by the motion classifier.

# Seniors' Council

## Risk of Biological Terrorism to Water Distribution Systems

*74548*

*S. J. Altman, M. Tezak, C. A. Souza, L. K. McGrath*


### Abstract

The goal of this research is to position Sandia as an expert on the transport and deactivation of biological agents through drinking water distribution systems. Water distribution systems are highly vulnerable to contamination and terrorism because there are virtually an infinite number of access sites to the systems downstream of the treatment sites. The intent of this research was to assess the impact of biofilms on the transport of biological agents. We ran laboratory scale experiments to better understand the adsorption and desorption of microorganisms and polystyrene microspheres onto, and from, biofilms. Results of experiments conducted to date show that colloids persisted in biofilms long after they were removed from the surrounding water under the conditions of our experiments. However, further experiments need to be run to evaluate these findings under different operating conditions. We developed a sound foundation to answer the questions initially proposed, even though we have not been able to conclusively determine how important biofilms are in controlling biocolloid transport. We will describe the capabilities of the biofilm laboratory to assess not only the impact of biofilms on drinking water systems, but also to study other biofilm related topics, such as biofouling of water treatment membranes. We will describe the experiments run to date to both grow repeatable biofilms from different biological sources and assess the integration of pathogens into these biofilms. Finally, we will describe our capabilities to visualize biofilms and the integration of pathogens in biofilms.

*Drinking water biofilm grown on a glass slide.*

### Accomplishments

We met our primary goal of developing the research capabilities to study the transport of biological agents through water distribution systems. We developed and demonstrated these capabilities through (1) growth of biofilms from our drinking water in drip flow systems, (2) growth of *Pseudomonas fluorescens* biofilms in annular reactors and drip flow systems, (3) experiments where "pathogens" are introduced to the annular reactor to determine how they interact with the biofilms, (4) development of methods to determine the concentrations of polystyrene microspheres and *Bacillus cereus* in the biofilms and reactor water, and (5) using a variety of methods to visualize biofilms. Development of these capabilities led to the funding of an FY 2006 LDRD project to study the decontamination of water distribution systems and led to a new separate project funded by the DOE desalination program to study biofouling.

We successfully grew biofilms from drinking water. To do this, we first dechlorinated the water by running it through two columns of granulated activated

carbon. We then mixed the drinking water with a solution containing nutrients and dripped it into a drip-flow reactor. With this high concentration of added nutrient solution, we were able to grow very thick biofilms.

Through a series of seven experiments, we demonstrated that we can grow repeatable *P. fluorescens* biofilms in annular reactors. We measured biofilm growth through heterotrophic plate counting. Once we developed confidence in our ability to grow repeatable biofilms, we ran a series of experiments where we introduced polystyrene microspheres and *B. cereus* (as a surrogate for anthrax) "pathogens" to the reactor. We collected biofilm samples over time to determine how well the pathogens integrate themselves in the biofilms. Our findings show that the pathogens are persistent in our biofilms over the time period of our experiments (as long as 35 days). The next step is to determine the impact of varying the shear force on pathogen integration in the biofilms to simulate flow in pipes. In order to conduct the pathogen introduction experiments, we developed a method to use gram selective agars to selectively count *P. fluorescens* and *B. cereus* in the biofilms and reactor water. We also developed methods to use fluorescence spectroscopy to measure polystyrene bead concentrations.

Visualization is an important component of biofilm related research. We investigated transmitted light microscopy, epi-fluorescence microscopy, confocal laser scanning microscopy, and hyperspectral imaging with multivariate analysis techniques (multivariate curve resolution). Each method has advantages and disadvantages. For example, both confocal laser scanning microscopy and hyperspectral imaging can provide three-dimensional images, but image collection time is much longer and more complicated than with epi-fluorescence microscopy. As a result of these experimental comparisons, we believe epi-fluorescence microscopy is adequate for much of the needs of our research.

# Developing Algorithms for Predicting Protein-Protein Interactions from Experimental Constraints

*75513*

*D. C. Roe, K. L. Sale*

## Abstract

Although it is now feasible to sequence entire genomes, there is still a large gap in obtaining functional information from these DNA sequences. This project addresses this gap by developing a new technology to go from primary DNA sequence to protein structure and function. This technology will extend the computational portion of Sandia's MS3D technology, a combined computational/experimental technique being developed to generate low resolution models of folded proteins, into a tool for understanding how proteins interact or "dock" with each other. The protein-protein docking problem, essential in understanding protein function, is a large unsolved problem that is related to protein folding. In practice, it has its own unique algorithmic challenges. Purely computational approaches to this protein-protein docking problem require highly accurate three-dimensional structures to be solved using x-ray crystallography (a slow experimental procedure) as a starting point. By taking an approach that combines computational docking with more readily available experimental constraints, we may be able to perform these docking calculations on less accurate homology protein structures that can be generated computationally from their DNA sequences rather than relying on x-ray crystallography. If successful, this tool will greatly speed and expand the number of protein-protein models that can be generated in a proteome. This will have numerous applications in both the pharmaceutical and biodefense communities. The goal of this project is to evaluate the feasibility of this approach and to develop a prototype protein-protein docking software tool that performs the docking calculations given the experimental constraints.

## Accomplishments

We focused on two aspects of protein-protein docking: (1) identifying the correct docking interfaces for a pair of proteins using bioinformatic derived information, and (2) algorithms to improve our ability to dock to homology based models using this information.

For the first part of the project, we shifted away from the original plan of using experimentally derived constraints (cross-linking) to guide protein-protein interaction in favor of bioinformatic constraints based on knowledge gleaned from protein-protein binding databases. The reason for this shift is that bioinformatic information about protein-protein binding is readily available. Therefore, algorithms that can exploit this information to identify protein-protein docking interfaces would have far more general applicability than algorithms that exploit experimentally derived constraints.

For the bioinformatics constraints, we expanded an approach we previously developed for identifying whether or not two proteins are likely to interact. In that study, we trained on a database of known interacting proteins, using products of "signature" descriptors based on sets of three amino acids as parameters, and a support vector machine as trainer. From the training set, we could predict which proteins interacted. For this study, we asked if these descriptors, parameterized from the large protein-protein interaction database, can also predict the docking interfaces on two interacting proteins.

The support vector machine for classifying protein interactions was trained on a set of 898 human proteins containing 1379 interacting pairs based on 1998 distinct experiments taken from the database of interacting proteins. Results from tenfold cross validation of the model gave an average prediction accuracy of 73.1%, which compares favorably with previous results. We then took a set of human protein complexes for which three-dimensional structures are available, extracted the amino acid sequences, split the sequences into domains consisting of 30 amino acids, and scored the domain interactions using the classifier trained on the human database of interacting proteins. In our first two test cases, we found good agreement between the domains predicted to interact and the actual domains as defined by changes in the exposed surface area.

For the second part of the project, prototyping docking algorithms, we started by completing an analysis of the degree of main-chain (protein polymer backbone) and side-chain flexibility that occurs during protein-protein docking to determine our priorities in

algorithm development. We compared the protein structures in the protein-protein complexes with their uncomplexed structures. We found that in a small fraction (approximately one-eighth) of cases there is significant main-chain modification on forming protein complexes, but in the majority of the cases, the major changes were in the side-chain rotamers. Side-chain placement becomes especially important when working with homology based protein structures where side-chain conformations are the least accurate part of the model; therefore, greater samples are needed from rotamer space. We decided to start with the known docking interfaces and focus heavily on side-chain placement. We tested several novel side-chain scoring systems and compared them to existing schemes in the literature. In addition, we developed initial algorithms to more thoroughly explore side-chain rotamer placements. Current approaches only sample ideal rotamers. We found from our surveys of side-chain positions that many vary far from their ideal rotamers to maximize van der Waals and hydrogen bonding interactions. For this reason, we chose to develop an algorithm to exhaustively sample all rotamer space that could easily be parallelized.

# Instrumentation Development for Real-Time Brain Wave Monitoring

*75514*

*L. F. Anderson, R. A. Salazar, M. K. Alam, B. Clough*

## Abstract

The human brain functions through a chemically induced biological process that operates in a manner similar to electrical systems. The device currently used in medical arenas to read brain electrical activity is the electroencephalogram (EEG), which is synonymous with a multi-channel oscilloscope reading.

Brain wave readings and recordings for medical purposes are traditionally taken in clinical settings such as hospitals, laboratories, or diagnostic clinics. The signal is captured via externally applied scalp electrodes using a semiviscous gel to reduce impedance. The signal will be in the 10 to 100 microvolt range. In other instances, where surgeons are attempting to isolate particular types of minute brain signals, the electrodes may actually be temporarily implanted in the brain during a preliminary procedure. The current configurations of equipment required for EEGs involve large recording instruments, many electrodes, wires, and large amounts of hard disk space devoted to storing large files of brain wave data that are then eventually analyzed for patterns of concern.

Advances in sensors, signal processing, data storage, and microelectronics over the last decade would seem to have paved the way for the realization of devices capable of real time external monitoring and possible assessment of brain activity. Myriads of applications for such a capability are likewise presenting themselves, including the ability to assess brain functioning and the level of functioning and malfunctioning.

Our plan is to develop the sensors, signal processing, and portable instrumentation package that could capture, analyze, and communicate information on brain activity of use to the individual, medical personnel, or others. We ultimately envision a system that would include a sensor detection mechanism, transmitter, receiver, microprocessor and associated memory, and audio and/or visual alert system. If successful in prototyping, the device could be considered for eventual implementation in application specific integrated circuit (ASIC) form or as a fully integrated complementary metal-oxide semiconductor (CMOS) microsystem.

## Accomplishments

We developed a handheld real time 32 channel brainwave data acquisition instrument that can acquire and store data at a 2500 Hz rate, and authored several basic brainwave data acquisition routines.

Signal levels from external scalp electrodes for normal brainwave activity are in the 10 to 50 microvolt range with seizure amplitudes in the hundreds of microvolts. We designed the bandpass analog front end circuitry to have a fixed voltage gain of approximately 2500, bringing the amplitude into a convenient voltage range for signal conditioning and signal processing. We simulated all front end electronic circuitry in PSpice to verify operation before breadboarding and eventual implementation on the printed circuit board. A bandpass frequency response of 0.5 to 100 Hz is accomplished with an eighth order filter design in the signal conditioning gain stages. The central controller chosen for all intelligent functions of the instrument is Phytec's phyCORE–MPC555 single board computer.

One of the goals was to receive and store 32 signal channels of EEG data as fast as the Nicollet BMSI model 6000, which has a 400 Hz data acquisition rate. Our instrument was able to acquire and store 32 channels at a rate of 2500 Hz. This leaves plenty of bandwidth for internal signal processing software techniques to be implemented and still maintain a system response greater than 400 Hz.

This prototype instrument performed well as a portable real time brain wave data acquisition system. Further testing with software that makes decisions based on the present conditions of a person's brain waves could be possible. Miniaturization of this unit would be an easy task even with the presently used integrated circuits and the same electronic design. Ultimately, this system could include a sensor, transmitter, receiver, microprocessor and associated memory, and audio and/or visual alert system. This device, through miniaturization, could be considered for eventual implementation in ASIC form or as a fully integrated CMOS microsystem. The possibility of a microsystem running a rigorous real time signal processing algorithm incorporated into a headband or baseball cap with a simple "early warning alert," be it an audio tone or visual alert, is not far off.

# Molten Salt Based Growth of Bulk GaN and InN

*80838*

*K. E. Waldrip, D. Ingersoll, J. L. Krumhansl, F. M. Delnick*

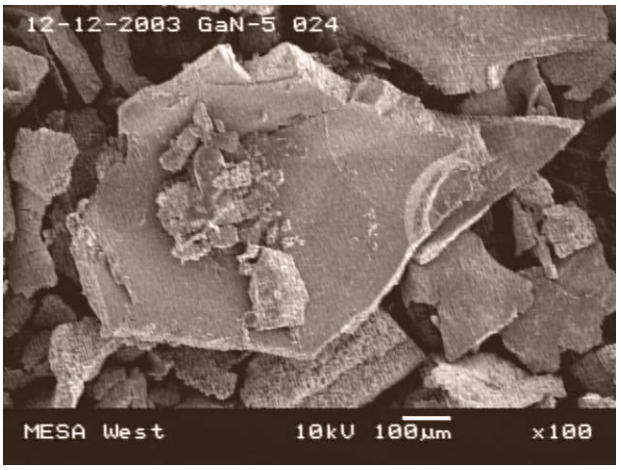
## Abstract

Gallium nitride (GaN) is a critical material for numerous advanced optoelectronic applications at Sandia, such as solid-state lighting and synthetic aperture radar. The greatest challenge facing development of this promising material system is the lack of widespread availability of low defect density, large area native substrates for epitaxial thin film growth. GaN poses substantial challenges for crystal growth in that it is a high melting point (m.p. > 2400 °C) refractory material that decomposes into gallium and nitrogen gas at 800 °C and 1 atmosphere, placing melt based growth techniques at the extremes of physics (> 60,000 atmospheres). Solvent based approaches also take place at high pressures, and the solubilities are low and kinetics slow.

Our goal is to develop a technique that circumvents the difficulties of other bulk growth techniques by precipitating the column III nitrides from a solvent, such as a molten chloride salt, that provides an excellent host environment for gallium nitride and indium nitride precursors. In particular, molten halide salts can solubilize both gallium ($Ga^{3+}$) and nitride ($N^{3-}$) ions, along with indium ions ($In^{3+}$), without reacting with them to the extent that they are no longer available for reaction with each other. Because the solvent also functions as an electrolyte, gallium and nitride ions can be produced in situ by electrochemical methods. Thus steady state growth may occur indefinitely, without solute depletion, by electrochemically producing $Ga^{3+}$and $N^{3-}$ ions at precisely the rate at which they are consumed by the growing crystal. This should, in principle, allow crystals of any size to be grown, with proper reactor design and engineering.

## Accomplishments

We performed initial experiments to determine the feasibility of one of several available pathways with this technique, namely, performing the initial solubility experiments of GaN in LiCl as a function of temperature. Solubility experiments are inherently difficult to do well; however, initial results tend to be very enlightening. Although the results contained a high degree of scatter, the data were found to lie above the ideal solubility curve predicted by an entropic approach to calculating solubility; that is, that the solute-solute interactions were equal to the solute-solvent interactions. There are two possible sources of the scatter. The GaN concentration in LiCl solution was measured by injecting a water based solution of GaN-containing LiCl into an inductively coupled plasma/mass spectrometer (ICP/MS). The GaN has a certain solubility in LiCl, but virtually none in water. It is therefore possible that the GaN agglomerated in water, causing spurious measurements. Another possibility is that the GaN did not dissolve molecularly in LiCl and was rather a dispersion to begin with, which would produce similar results. It is worth pursuing additional work to determine the solvation mechanism for GaN in LiCl.




*Scanning electron microscopy image of 1 mm diameter GaN crystal grown at atmospheric pressure in one afternoon.*

We designed and constructed the electrochemical apparatus for performing electrolysis. We explored the electrochemical reduction of nitrogen in LiCl-KCl and found it to proceed readily on nickel electrodes at 400–500 °C at a voltage of 200–300 mV positive of $Li^{+}$/Li. We observed no dependence on gas flow rate at the flow rates used in this study.

## Refereed

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and J.Y. Tsao, “Atmospheric Pressure Molten Salt-Based Growth of Bulk III-N’s,” presented at the Electrochemical Society, Honolulu, HI, October 2004.

A.M. West, “Effect of Dislocations on AlGaN/GaN High Electron Mobility Transistors,” University of Florida Doctoral Thesis, University of Florida Press, Gainesville, FL, May 2005.

## Other Communications

K.E. Waldrip, T.M. Kerley, and J.Y. Tsao, “Molten Salt-Based Growth of Bulk GaN and InN,” presented at the 2nd International Indium Nitride Workshop, Kailua-Kona, HI, January 2005.

# Micropower Chemical Fuel-to-Electric Conversion: A "Regenerative Flip" Hydrogen Concentration Cell Promising Near Carnot Efficiency

*84315*
*K. Wally*

## Abstract

The purpose of this project was to conduct an exploratory investigation of a micropower converter based on an electrochemical hydrogen concentration cell operating between source and sink metal hydride beds. The source/sink relationship of the beds is cyclically established by operating the source at a higher temperature than the sink. As the source becomes depleted of hydrogen and the sink fills, the heating and cooling can be reversed, the concentration cell electrodes can be commutatively reversed, and operation can continue with hydrogen flow reversed. The resultant regenerative use, but not consumption, of hydrogen is characteristic of the converter and reflected in the "regenerative flip" name. We conducted experiments to confirm hydrogen concentration cell performance, to demonstrate satisfactory performance of a metal hydride bed, and to investigate the effect of internal resistance on micropower performance.

## Accomplishments

We conducted a series of experiments to explore the key elements of performance for the proposed micropower converter concept.

To conduct hydrogen concentration cell experiments, we employed a small unit cell hydrogen fuel cell apparatus that we modified to provide added support to either side of the membrane electrode assembly (MEA). This accommodated the differential pressures used in the concentration cell operation. Lecture bottles with regulators to adjust operating pressure supplied source and sink hydrogen. We used two different MEAs: one based on a Nafion 112 membrane (51 microns thick) and the other based on a Nafion 117 membrane (183 microns thick). The MEAs consisted of identical gas diffusion electrodes of the ELAT type (electrode sponge of platinum catalyst on carbon powder heat-bonded with Teflon powder and Nafion powder on a carbon cloth substrate) on either side of the Nafion membrane, with the electrodes heat-bonded to the Nafion using a Nafion-alcohol solution to "butter" the interface before heat/pressure bonding. No intentional additional humidification of the hydrogen gas was provided. We measured gas pressures in mm of Hg using a manometer apparatus.

In order to achieve and demonstrate adequate initial conditioning, we first operated the cells as hydrogen/oxygen fuel cells. We established open cell voltages and then switched a series of increasing load resistances into the circuit. We also measured voltage and current under load. We used fuel cell mode operation to estimate the internal resistance of the cell from the slope of the polarization curves measured under the resistive loads. Once we demonstrated typical hydrogen/oxygen fuel cell performance for a given MEA and Nafion membrane thickness, we purged the oxygen cathode and applied hydrogen to this electrode to complete the hydrogen concentration cell configuration. We measured open cell voltage and then measured a series of voltages and currents using the series of increasing load resistances. Each MEA thickness operated in both forward and backward directions (they are identical), with electrodes reversing their roles of anode and cathode. The cells operated at several differential hydrogen pressures corresponding to palladium hydride plateau pressures at selected source and sink operating temperatures. We achieved peak open cell voltages of 51 mV for the N112 membrane and 47 mV for the N117 membrane. Results correspond well to the calculated value of 57 mV, confirming the efficacy of the hydrogen electrodes. Peak power for the N117 membrane (0.45 ohm internal resistance) was about 1.4 $mW/cm^2$ at 25 mV load voltage and 55 $mA/cm^2$ current. Peak power for the N112 membrane (0.2 ohm internal resistance) was about 3.1 $mW/cm^2$ at 25 mV load voltage and 125 $mA/cm^2$ current.

We conducted a series of pressure composition temperature (PCT) experiments using a small palladium hydride bed in communication with a small calibrated volume. Thermocouples measured temperatures. We hydrided and dehydrided the palladium hydride several times until we obtained well conditioned PCT results (comparable to previous PCT results for similar specimens). We generated an absorption curve, followed by a desorption curve, and repeated. We then changed the configuration to add communication with

a Nafion membrane. We maintained the humidity of the Nafion membrane by contacting the backside of the membrane with wet cotton balls. Again, we generated absorption, then desorption PCT curves, and repeated. We observed no discernible effect from the presence of water vapor (approximately 45% relative humidity).

We were unable to complete the integrated device demonstration test we had planned. Nevertheless, the above results substantially confirm the original hypothesis, and an integrated device would most likely operate as intended. The concept appears as if it could be engineered to produce small milliwatt micropower converters.

# MEMS Based Arrays of Micro Ion Traps for Quantum Computation and Quantum Simulation Scaling

*85513*
*M. G. Blain, J. G. Fleming, C. P. Tigges*

## Abstract

The mathematical basis for quantum processing is relatively well established, but the physical means for implementation is sorely lagging. Quantum information processing algorithms for factoring and searching provide great gains in computational complexity over classical algorithms. Separate from these information processing applications, we expect to achieve significant advances in the ability to simulate quantum processes by using quantum devices to perform the quantum simulation (QS). Several technologies are potential contenders for performing practical quantum processing operations, but all of these technologies suffer from engineering problems in scalability and control.

Ion trap techniques are among the most promising of approaches and constitute a fertile area of research. However, few research organizations are able to fabricate them, much less fabricate the entire arrays of ion traps that would be required to perform meaningful quantum processing operations. The purposes of this effort are to (1) demonstrate viable ion trap array fabrication techniques, and (2) collaborate with external partners to validate the utility of these arrays.

## Accomplishments

We designed, simulated, and fabricated planar ion trap electrode structures that are micrometer in scale, including one that is multiplexed for discrete control of individual ion trap segments. We designed these ion traps for application in trapped ion QS experiments in collaboration with Dana Berkeland at Los Alamos National Laboratory (LANL). We took advantage of the design and processing knowledge established in prior LDRD funded efforts to microfabricate massively parallel arrays of three-dimensional (3-D) cylindrical ion traps (CITs) as micro mass analyzers. In addition, we simulated operation of the ion trap electrode arrangements in order to validate trap functionality prior to fabrication. We designed and simulated, microfabricated, and released ion traps that are being packaged for QS experiments at LANL. These all new planar ion trap designs, including Paul-Straubel and segmented linear traps, provide the high conductivity and low capacitance radio frequency requirements necessary to trap ions for QS experiments. The linear traps are segmented so that they allow the shuttling of ions (qubits) between trap segments to perform discrete ion state manipulations. An important feature of these ion traps is the through-substrate optical access for the laser light used to manipulate the electronic and translational eigenstates of the ions.

In addition to the work on ion traps, we made preliminary experimental measurements of the minimum actuation time of existing silicon microelectromechanical systems (MEMS) based tilting mirrors in order to understand their applicability as fast microoptic devices in QS and quantum computation (QC) experiments. Measurement results show that 120x180 square-micrometer micromirrors, fabricated using Sandia's SUMMiT V™ (Sandia Ultra-planar, Multi-level MEMS Technology 5) silicon surface MEMS process, can be switched from -10 to +10 degrees in less than six microseconds. This is an order of magnitude faster than previous results for these mirrors. Since these mirrors were designed for a very different application, one not requiring actuation, an effort was initiated to design new silicon based MEMS micromirrors with actuation times of less than one microsecond. Initial design analyses show that there are new approaches for actuation of smaller mirrors that will allow sub-microsecond switching through angles relevant to QS and QC experiments.

This project enabled Sandia to take advantage of a unique opportunity to establish a role in the engineering of hardware necessary for QC and QS. We were also able to answer some critical questions regarding the scalability of a QC. A QC is a much more powerful device, in principle, than any existing or future classical computer because the superposition principle allows an extraordinarily large number of computations to be performed simultaneously. Certain problems believed to be intractable on any existing or future conventional computer could be efficiently solved using this quantum parallelism. Problems such as integer factorization and discrete logarithms form the foundation

of nearly all publicly used encryption techniques; thus, a QC would enable quantum cryptography and quantum key distribution. Quantum information science in general, and QC in particular, will likely be the basis for future information security technologies (thereby addressing fundamental national security and intelligence problems) and future QS and modeling capabilities (thereby addressing computational and information science problems related to our nuclear weapons mission). This project increased our knowledge base in quantum information science and is facilitating a growing program, presence, and role in the engineering of quantum devices.

# Diffusionless Fluid Transport and Routing Using Novel Microfluidic Devices

*89669*

*D. Reichmuth, E. B. Cummings*

## Abstract

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., using classical test tubes and beakers or more modern robotic pipetters and microwell plates). Current microfluidic devices typically employ 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 along the length of the channel and generally across the branches of the network.

We are demonstrating the feasibility of on chip devices 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 multi-step 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. 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.

## 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 glass chip. Currently, we are using glass microchips to test valve materials and segmenting fluids. We are also examining the use of polymer based microchips because they could be cheaper and quicker to manufacture.

Our tests with existing glass chips from a previous project show promise: we demonstrated 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 two-phase packet generation. We made substantial design changes for this project's chips and generated the photomasks. The initial batch of glass chips produced at Sandia had significant defects due to errors in the fabrication process. While some chips are usable from this batch, it is likely that a new batch will be required. We are investigating using outside contractors for the glass chip fabrication, and stamps for embossed plastic chips have been finished.

Despite the fabrication difficulties, we established that, once generated, packets can be routed on-chip using simple off-chip valving. Also, these chips are the first to include particulate filters between the inlets and the valve. To date, we have not had particles foul the valve element; therefore, we significantly increased valve lifetime.

We are also taking advantage of the ability to create segmented flow using Sandia designed microfluidic connectors. These connectors allow the testing of passive segmentation schemes. For example, we demonstrated that an analyte peak can be segmented by a fluorinated hydrocarbon. The segments can be analyzed for an arbitrary amount of time. We demonstrated that we can extend the analysis time by a factor of 5, which has the potential to greatly increase the signal to noise ratio. Also, diffusion between packets cannot occur, so long term storage of nanoliter-sized volumes is possible.

## Refereed

D.S. Reichmuth and E.B. Cummings, "Segmentation for Enhanced Detection, Storage, and Routing of Analytes," presented and in *Proceedings of the MicroTAS 2005 Technical Digest*, Boston, MA, October 2005, vol. 1, CD-ROM.

## Other Communications

D.S Reichmuth and E.B. Cummings, “Active and Passive Segmentation for Transport and Storage of Analytes,” presented at the Gorden Research Conference, Oxford, UK, July 2005.

# Emulsion Technology for Sample/Contaminant Collection

*90498*

*C. J. Bourdon, M. E. Welk, C. A. Souza*

## Abstract

The purpose of this project is to study a novel emulsion based 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 will be 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. Due to the ubiquitous affinity for adsorption to interfaces, the emulsion pre-concentrator can be applied to a broad spectrum of biological and chemical materials of interest in homeland security and water surety applications.

This study will investigate the promising capability of emulsions to achieve high levels of sample and contaminant collection for a wide variety of potential chemical and biological targets. Initial studies will focus on demonstrating the feasibility of the emulsion collection concept; then on examining basic technological challenges for each stage of the process: emulsification of the mixture, diffusion, and trapping on the interface; then on combining the emulsion droplets for concentration and collection. We will then proceed to focus on resolving issues involved in incorporating emulsification, collection, and coalescence into a single system. This emulsion based collection technology offers great promise to significantly improve the sensitivity, efficiency, and applicability of current collection and detection systems. The advantages of our approach are clearly greatest for collecting very dilute quantities of a contaminant and would benefit many Homeland Security and Department of Defense applications.

## Accomplishments

We made rapid progress starting up laboratory experiments to explore the fundamental physics required for an emulsion pre-concentrator. So far, we focused our efforts in two areas: trapping of materials at heterogeneous emulsion interfaces and understanding fundamental dynamics of emulsions.

To explore the behavior of various classes of materials that may be of interest for homeland security applications, we experimented with the adsorption of biological cells (purified chicken blood), particles (fluorescent polymer 0.1 micron beads), and organic molecules (fluorescent Rhodamine dye) to a stationary oil-water interface. Water with a known concentration of the target species is placed in a vessel, and oil is added to cover the surface of the water. At measured intervals, samples are taken from the water phase. A spectrophotometer reveals that the biological cells and the organic molecules adsorb quickly to the interface; experiments are ongoing with the nanoparticles. Preliminary quantitative results for the organic molecules show a decrease in the dye concentration from $4.2 \times 10^{-12}$ M to $2.6 \times 10^{-12}$ M within the first 10 minutes of contact with the oil. No desorption or additional adsorption of the Rhodamine dye occurs up to 3 days after the oil was added. This decrease in concentration equates to roughly $9 \times 10^{9}$ molecules per $cm^2$ of the stationary oil-water interface. In an emulsion with both a much greater interfacial surface area and reduced distance for the target species to diffuse through, more molecules should adsorb even more rapidly.

We also implemented an experimental system to study the formation, motion, and coalescence of drops in a controlled emulsion. We were able to create drops ranging in size up to 5 mm in diameter with a model system of silicone oil and water. Unfortunately, since the coalescence dynamics are very sensitive to external contamination, it has been very difficult to get reproducible results in our current lab space. We are working to move this experimental system into a cleanroom. In the interim, we decided to explore some other methods for examining the system dynamics. Using the formation process for a drop, we can perform real time analysis of the drop shape to measure the adsorption/desorption of materials into the interface through changes in the interfacial tension. This will complement the quantitative measurements of adsorption discussed above. We will also use system level measures of coalescence times to characterize systems rather than looking at individual drop dynamics.

# Visualizing Higher Order Finite Elements

*90499*

*D. C. Thompson, P. P. Pébay*

## Abstract

Finite elements are used to create piecewise polynomial approximants to the solution of partial differential equations for which no analytical solution exists. These polynomials represent fields such as pressure, stress, and momentum. In the past, these polynomials were linear in each parametric coordinate. Each polynomial coefficient must be uniquely determined by a simulation, and these coefficients are called degrees of freedom. When there are not enough degrees of freedom, simulations will typically fail to produce a valid approximation to the solution. Recent work shows that increasing the number of degrees of freedom by increasing the order of the polynomial approximation (instead of increasing the number of finite elements, each of which has its own set of coefficients) can allow some types of simulations to produce a valid approximation, with many fewer degrees of freedom than increasing the number of finite elements alone. However, once the simulation determines the values of all the coefficients in a higher order approximant, tools do not exist for visual inspection of the solution.

This project focused on a technique for the visual inspection of higher order finite element simulation results based on decomposing each finite element into simplicial regions where existing visualization algorithms, such as isosurfacing, will work. The requirements of the isosurfacing algorithm are enumerated and related to the places where the partial derivatives of the polynomial become zero. The original isosurfacing algorithm is then applied to each of these regions in turn.

## Accomplishments

Our main goal was to examine how the parametric domain of a higher order (i.e., nonlinear) scalar field, defined over a finite element, could be decomposed into regions where the assumptions of the existing (linear) isosurfacing algorithm hold. Although we previously showed that these regions were linked to the critical points of the field, where all partial derivatives vanish, we did not know how to produce a simplicial decomposition of the domain that would guarantee the existing isosurfacing algorithm would work. We developed an algorithm to do just this, along with proof that it will terminate as long as the critical points are isolated. For non-isolated critical points, the algorithm may not always terminate, but limiting the number of times the initial decomposition is subdivided will provide a piecewise approximation of the locus of non-isolated critical points.

In addition to meeting our final goal, we also examined the properties of several different polynomial solvers used to find isolated critical points in one, two, and three dimensions. We tested homotopy solvers (which slowly transform a polynomial with known roots into the given polynomial and trace the paths that the roots take during the transformation), resultant based solvers in one and two dimensions (which construct a matrix determinant whose eigenvalues are roots of the polynomial and whose condition numbers are used to discriminate valid roots from synthetic roots introduced by approximation error), and deflation based solvers in one dimension (that find low order roots and divide them from the existing polynomial).

## Refereed

W. Schroeder, F. Bertel, M. Malaterre, D. Thompson, P. Pébay, R. O'Bara, and S. Tendulkar, "Framework for Visualizing Higher-Order Basis Functions," to be published in *Proceedings of the 2005 IEEE Visualization Conference.*

# Bioagent Detection Using Miniaturized NMR

*90506*

*T. M. Alam, D. P. Adams, J. D. Williams, H. Fan*

## Abstract

This project is directed toward the development of a miniaturized nuclear magnetic resonance (NMR) detector utilizing advances in two major areas. The first component of the proposed research involves the miniaturization of the detector system. By utilizing the microelectromechanical systems fabrication expertise here at Sandia, we will be able to place this NMR device on a chip with microfluidic capabilities. The second component of the project involves the development and refinement of the nanoparticle amplification process. The basis for this detection amplification employs the combined use of biological ligands (antibodies) labeled with paramagnetic nanoparticles. This device will allow for the reliable detection of bioagents in a range of sensing environments.

## Accomplishments

We made significant progress in FY 2005.

(1) We fabricated the NMR microcoils on two different platforms. We presently have these devices ranging from 500 µm down to 10 µm. Additional testing of these NMR devices is ongoing.

(2) We also fabricated a microcoil on a cylindrical capillary. The initial design was 500 µm in diameter, but subsequent designs are proposed down to 80 µm diameter coils.

(3) We obtained the first NMR signal for the cylindrical device at low magnetic fields (1 T). While there has been significant work on microdevices at high magnetic fields, this represents the first NMR microcoil operating at low magnetic fields. The low field will allow for future miniaturization of the entire device by use of chip fabricated magnet systems.

(4) We accomplished the synthesis of paramagnetic nanoparticles with controlled particle size and distributions. This work includes the modification of the surface to assure water solubility. Conjugation of these novel nanoparticles with biological antibodies is ongoing.

(5) By utilizing commercially available superparamagnetic iron oxide nanoparticles (SPIONs) conjugated to the anti-PSMA antibody 3C6, we demonstrated the dramatic amplification of water relaxation effects within the cylindrical 500 µm NMR coil.

## Other Communications

A. McDowell, E. Fukushima, L. Sillerud, and T.M. Alam, “Low-Field Micro-Coil Probe Development for Portable NMR,” presented at the 8th International Conference on Magnetic Resonance Microscopy, Utsunomiya, Japan, August 2006.

# University Collaborations

## Friction in Micromachine Interfaces

*57308*
*B. R. Antoun*

### Abstract

The University of California at Berkeley developed polysilicon micromachines designed for the purpose of studying the tribological properties of microelectromechanical systems (MEMS) such as interfacial adhesion and friction. Understanding the physics controlling the interfacial behavior is critical to the reliability and efficiency of micrometer scale devices. At this small scale, the surface forces can be on the same order of magnitude as the inertial forces, and therefore cannot be ignored. The purpose of this research is to establish experimental protocols to elucidate the tribological behavior of MEMS devices.

The research includes four specific areas of study that will be conducted at the University of California at Berkeley. The first experiments involve measuring the adhesion forces for microstructures in contact at known external contact loads. A parametric study is performed at a variety of important environmental conditions, including high vacuum and high relative humidity. The second study is an attempt to measure the static friction forces of microstructures at known external contact loads. These friction forces are then combined with measured adhesion forces to elucidate a true normal load and thereby a true coefficient of friction. The third portion of the project is to measure the dynamic friction behavior of microstructures experiencing sinusoidal sliding contact. Finally, experiments will be performed in order to interpret the wear behavior of the contacting microstructures under different contact loads and environmental conditions.

### Accomplishments

We designed a surface micromachined test structure specifically for studying the sidewall adhesion characteristics of MEMS devices. We investigated the dependence of adhesion on contact load under quasi-static loading conditions. We found in this study that the adhesion force is linearly dependent on the contact load due to the increase of the real contact area and the decrease in the separation distance of the non-contacting asperities. We obtained insight into the relative contributions of van der Waals and capillary forces to the measured adhesion force and apparent work of adhesion. We effectively eliminated several shortcomings of previous adhesion studies of MEMS, and we accomplished the true pull-out force under different environmental testing conditions. The experimental methods developed enable the isolation of the van der Waals component of the adhesion force and the determination of the contributions of contacting and non-contacting asperities to the total adhesion force. We performed a complete parametric study of sidewall adhesion and established a set of solid experimental methods.

We designed a second test structure for testing the static and dynamic friction characteristics, as well as the wear properties, of MEMS devices. Initial data from these devices show that we will gain insight into the effect of contact load and environmental conditions on the friction behavior. The experiments will also take into account the relatively high adhesion forces in MEMS devices (that can be on the same order of magnitude as the external normal loads) in order to obtain a more thorough understanding of the contact mechanics at the interface.

# Lipid Microarray Biosensor for Biotoxin Detection

*57309*
*A. K. Singh*

## Abstract

We present a technique to create micron sized lipid bilayer arrays on flat substrates and within microfluidic channels that can be used for multiplexed sensing of analytes. The arrays used to demonstrate the technique consist of pattern supported lipid bilayers (SLBs) populated with ganglioside receptors. To pattern the SLBs, we used a polymer liftoff technique that, after lipid deposition, revealed SLB arrays containing either ganglioside GT1b or GM1. The ganglioside receptors selectively bind either cholera toxin subunit B (CTB) or tetanus toxin fragment C (TTC). Microfluidic connections to independent channels etched into silicon substrates allowed preparation of lipid domains with different receptors to achieve segregation and selective binding from a binary mixture of the toxin fragments in one device. We detected toxin fragment binding by epifluorescence down to ca. 20 pM and 100 pM concentrations for TTC and CTB, respectively.

## Accomplishments

We demonstrated that ganglioside populated SLB arrays can be patterned both on planar substrates and within microfluidic channels with a polymer liftoff technique. This technique can be easily extended to generate protein or deoxyribonucleic acid (DNA) arrays, as well. We also demonstrated that the patterned SLBs containing gangliosides can bind toxin fragments selectively and segregate them from a binary mixture. The patterned SLBs, once the polymer is removed, are stable and functional over a long period of time when stored under the appropriate conditions. The limit of detection for toxin fragments bound to the patterned arrays is in the picomolar range for optical detection. This suggests the possibility to use these patterned arrays for biosensor applications coupled with on-chip detection schemes such as evanescent wave fluorescence, diffraction sensing, electrochemical signaling, voltammetry, and piezoelectric sensing.

We used substrates to pattern SLBs into 0.5 to 76 micron features with square and rectangular shapes. We incubated lipid vesicles with plasma treated substrates and allowed them to form SLBs in the exposed areas of the substrate. We could only achieve patterning of lipid domains with different compositions on planar substrates through the use of a physical barrier such as a poly(dimethylsiloxane) (PDMS) well. The need for a physical barrier derives from the change in hydrophilicity of the Parylene coating after plasma treatment. This patterning procedure amounted to spotting in a similar fashion to DNA microarrays, but had to be kept in fluid at all times to avoid desorption.

We used planar substrates to determine the characteristics of the patterned SLBs. We characterized lipid patterns obtained after polymer liftoff by epifluorescence, fluid cell-tapping mode atomic force microscopy (FC-TMAFM), and fluorescence photobleach recovery (FPR). We first tested patterned lipids for the formation of SLBs. Epifluorescence images verified the presence of patterned lipids, but did not allow complete certainty as to the presence or absence of a bilayer. Thus, we used alternate methods to corroborate the formation of lipid bilayers.

We performed binding assays to determine the optical limit of detection and binding specificity for the SLB arrays. We used concentrations ranging from 1 mg/mL–100 ng/mL in successive dilutions and epifluorescence images taken to assess the detection limit. The lowest detectable concentration showing a discernible pattern was ca. 100 pM (incubating with 100 µL at a concentration of 1 µg/mL) in the binding assays for the CTB fragment and ca. 20 pM (incubating with 100 µL at a concentration of 1 ug/mL) in the binding assays for the TTC fragment. We could not discern lower concentrations (100 ng/mL) of either toxin fragment as complete patterns, nor could they be recovered after image processing.

We used the substrates containing etched trenches and patterned SLB arrays with different lipid compositions to segregate toxin fragments from binary mixtures. We optimized the operational pH of the mixtures and found it to be 6.5. This allowed us to obtain specific binding of the toxin fragments to their corresponding gangliosides and to achieve segregation from the binary mixture within the trenches with low nonspecific binding. Incubation of the patterned microfluidic substrates with a single toxin fragment yielded similar results as those obtained with planar substrates.

These results show the capability of this patterning technique to generate patterns of biomolecules within microfluidic channels. They show that the patterned SLBs containing different ganglioside composition are capable of segregating the toxin fragments present in a binary mixture. They also show that we can extend the procedure to include more than two types of receptors to have a larger number of toxins specifically bound on a single chip. The possibility of patterning arrays within microfluidic channels offers the opportunity of integrating detection schemes to microfluidic devices, with areas of detection confined to the patterned arrays. Furthermore, we can use all the advantages of microfluidic devices, such as low volume requirements and multiplexing capabilities, with biosensing arrays.

## Refereed

J.M. Moran-Mirabal, J.B. Edel, G.D. Meyer, D. Throckmorton, A.K. Singh, and H.G. Craighead, "Micrometer-Sized Supported Lipid Bilayer Arrays for Bacterial Toxin Binding Studies Through Total Internal Reaction Fluorescence Microscopy," *Biophysical Journal*, vol. 89. pp. 296–305, 2005.

# Advanced Manufacturing Techniques Using Rapid Prototyping

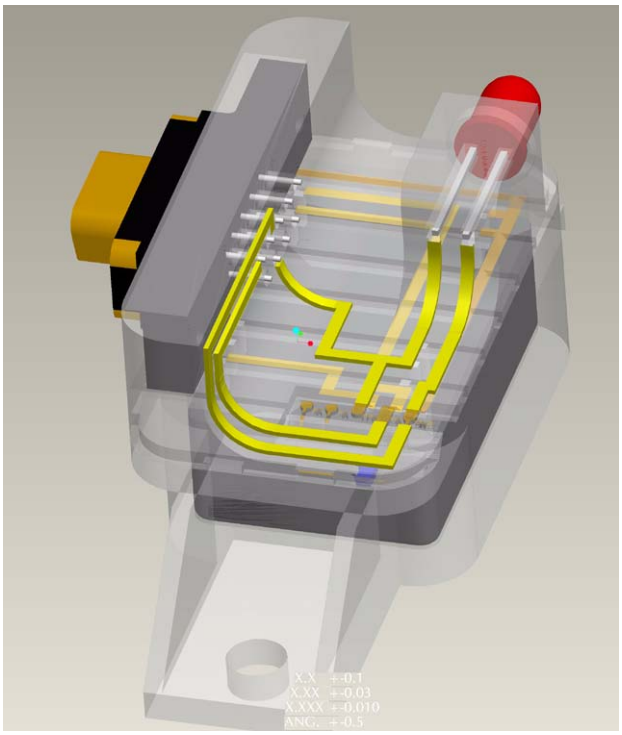
*57310*
*J. A. Palmer*

## Abstract

The focus of this effort is the use of the 3D Systems' Viper si2 stereolithography apparatus (SLA) rapid prototyping (RP) machine for the direct manufacturing of mesoscale parts (with features on the order of microns to millimeters) and assemblies in a variety of applications. These include microfluidic components and mesoscale discrete electrical components such as switches and coils. The capability to produce this class of devices expands the rapid responsive infrastructure to manufacture chem/bio sensors and other miniaturized national security products. This research will build on previous success in the manufacturability of novel mesoscale parts and assemblies, and ultimately yield new multiprocess, multimaterial SLA-based RP platforms that move beyond the capabilities of current systems. The past fiscal year saw dramatic achievements in the area of integrated rapid manufacturing with the introduction of the world's first integrated direct write (DW) SLA system. We also constructed a novel multimaterial SLA. We constructed successful prototypes in support of Sandia's Rapid Prototyping of High Density Circuitry (RPHDC) effort.

## Accomplishments

We conducted research as part of the University of Texas at El Paso (UTEP) partnership in FY 2005. We achieved two noteworthy achievements. First, UTEP constructed and operated the world's first integrated direct write dispensing and stereolithography apparatus. This prototype was supported by surplus 3D Systems' stereolithography equipment provided by a grant from Sandia and the National Science Foundation. We rolled out a novel multimaterial SLA. The multimaterial system includes a carousel of complementary resin materials with an intermediate cleaning step that we initiate under computer control. These prototypes are the fruit of two joint patent applications filed as a direct result of the partnership. We applied UTEP's expertise in RP and RPHDC techniques to develop a successful satellite communications system power supply, multifunctional smart structure that featured encapsulated electronics and integral solderless three-dimensional DW interconnects. Smart structure characterization consisted of thermal cycle and vibration resistance testing that culminated in greater than three refereed publications. UTEP provides ongoing engineering support and consultation on numerous Sandia programs and proposals. Most recently, UTEP aided in Sandia's demonstration of novel three-dimensional LIGA (for the German term *Lithographie, Galvanoformung, und Abformung*, for lithography, electroforming, and molding) technology assisted by microstereolithography. Future work includes a Lockheed Martin Shared Vision Program proposal involving RPHDC applications, and other work-for-others programs.



*Smart structure with encapsulated electronics with 3-D Direct Write (DW) interconnect.*

## Refereed

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## Other Communications

R.B. Wicker, A.V. Ranade, F. Medina, and J.A. Palmer, "Embedded Micro-Channel Fabrication Using Line-Scan Stereolithography," to be published in *Assembly Automation Special Issue: Rapid Protoptying.*

J.A. Palmer, B. Jokiel, C.D. Nordquist, B.A. Kast, C.J. Atwood, E. Grant, F.J. Livingston, F. Medina, and R.B. Wicker, "Miniature RF Components Enabled by Mesoscale Rapid Manufacturing," to be published in *Rapid Prototyping.*

# Advanced Techniques for Multi-Objective Discrete Optimization

*66450*

*J. E. Campbell, S. H. Jacobson, G. K. Kao*

## Abstract

A wide variety of real world, practical applications can be modeled as discrete optimization problems. Many of these problems are intractable and require specialized algorithms and heuristics to obtain even near optimal solutions in a reasonable amount of computing time.

Two common characteristics of difficult, real world optimization problems are (1) hundreds or even thousands of decision variables that result in a massive decision space, and (2) the need to optimize multiple objective functions.

Large input spaces are common to many problems, such as maximizing system performance or minimizing logistics footprint. While such problems often have large numbers of input variables, many of these variables may have little effect on optimization objectives. Furthermore, variables that do affect key objectives may do so only in limited regions of their ranges. The result is often a massive input space in which promising search regions are extremely small. To address this issue, this research will examine adaptive learning techniques that can help focus an optimization search on promising areas of the decision space. The approach will examine techniques to focus optimization searches on input regions that are likely to contain useful solutions.

To address the second problem area, the need to optimize multiple objectives, this research will examine algorithms for obtaining Pareto optimal solutions and procedures to understand the trade-offs between these solutions. We will then use such results for post-optimal analysis to obtain sets of the “best of the best” Pareto optimal solutions. Given that discrete optimization problems cover a wide range of applications, the research conducted in this area has numerous potential applications.

This work seeks to fill an important need for optimization techniques that can be applied to complex systems-of-systems such as Future Combat Systems (FCS). FCS performance is evaluated in terms of several measures of effectiveness (MOEs). Optimizing FCS performance across multiple MOEs and multiple platform families presents a challenge that is beyond currently used optimization techniques.

## Accomplishments

One of the accomplishments of this project over the past year is the significant research progress in the area of post optimality analysis. We addressed the problem of identifying the best subset of solutions across very large sets of Pareto optimal solutions obtained from any general multi-objective optimization techniques. We formulated a new discrete optimization problem for obtaining such optimal subsets of Pareto solutions. We implemented an enumeration algorithm to solve the discrete optimization problem generating the optimal preferred subsets of Pareto solutions. We implemented neighborhood search heuristics, as well as greedy elimination heuristics, to address the efficiency of solving the discrete optimization problem. We created three example problems to compare the performance of the enumeration algorithm with the two different heuristics.

Another area of research involves initial examination of methods for identifying and eliminating decision variables that have little or no effect on optimization objectives. We used a genetic algorithm as the general optimization procedure. Previously, we calculated partial rank correlations between input variables and objective values of a stratified sample of initial solutions. A pre-defined threshold cutoff correlation determined which decision variables to eliminate. This reduced the size of the input decision space by several orders of magnitude.

We implemented two new approaches to further address the problem of reducing the size of the input variable space. The first approach is an iterative procedure incorporated within the genetic algorithm framework. After a fixed number of generations, we used the top solutions found to determine the new reduced ranges associated with the decision variables. This process will incrementally reduce the size of the input variable space for the following generations. The second approach used a stratified sampling technique, which ensured a good coverage of the input variable space to generate an initial population. We used a ranking scheme across input variables and objective values to determine the significance of each input variable. The input space is then reduced prior to the optimiza-

tion procedure. We incorporated both approaches in genetic optimization, an application developed at Sandia.

## Other Communications

G. Kao and S. Jacobson, "Finding Prefer Subsets of Pareto Optimal Solutions," submitted to *Journal of Optimization Theory and Application.*

# Non-Radioactive Safety & Performance Issues with Supercritical Water (SCW) Reactor Safety Technologies

*66809*
*J. V. Zuffranieri*

## Abstract

Operating water at supercritical conditions in a light water reactor (LWR) can increase its efficiency by as much 30% above current conditions. As water increases in temperature through the pseudo-critical point, it undergoes large property changes that can significantly alter heat transfer. Current models used to predict heat transfer near or at the supercritical region do not include these property variations and thus are not valid for these conditions.

The University of Wisconsin has an operational supercritical water (SCW) flow loop used for corrosion studies and the characterization of materials for use in SCW reactors. It is our intent to make use of this existing facility to perform detailed measurements of heat transfer in the supercritical regime. To accomplish this we focused on the following six tasks:

(1) Modifying the existing SCW facility to allow for heat transfer studies on an internal high heat flux heater rod.

(2) Developing an optical laser Doppler velocimetry (LDV) system capable of measuring local flow velocities with frequency components up to 3 kHz next to the simulated heated fuel pin with SCW conditions (25 MPa, 500 °C).

(3) Developing an optical Rayleigh scattering system to measure discrete point density fluctuations with frequency content comparable to that of the velocity components.

(4) Developing a database of heat transfer, axial and radial velocity measurements under a variety of flow, temperature and heat flux conditions using internal heater thermocouples and particle image velocimetry (PIV).

(5) Developing a source term based on cross-correlations of fluctuating density and velocity components to augment the standard KMODEL and use FLUENT to compare computational fluid dynamics simulations with experimental results.

(6) Comparing previously proposed correlations for supercritical heat transfer with our experimental and computational results, and recommending modifications to those correlations to properly describe supercritical water heat transfer.

## Accomplishments

Experimental design modifications to the SCW loop are nearing completion. Key modifications include an optical test section, a heater rod, and a cooling system.

We designed a test section that contains two orthogonal sapphire windows with a 2 square cm view area. This allows optical access for measuring fluctuating velocities and densities in the radial direction from the boundary layer of the heater rod wall to the bulk fluid. In order to make optical access feasible, we chose a square flow channel. This permits flat optical windows that will not disrupt flow and allows for easy index of refraction corrections with measuring instrumentation. We performed stress calculations with the relevant software to verify the feasibility of the design. We acquired the necessary materials, and machining is currently under way.

We used an in-depth literature survey to develop a set of experimental conditions to be investigated. Because it was desirable to operate at a variety of heat flux and mass flux conditions, we needed a new cooling system. The new cooling system developed will allow us to operate at any heat flux and mass flux attainable with our heater rod and SCW pump. The cooling system is made up of eight copper cooling coils of different lengths tightly wrapped to the main Inconel piping. A booster pump supplies building water to the cooling coils at flow rates up to 30 gallons per minute. A manifold system controls flow rates and isolates cooling lines, allowing for a variable heat removal.

# Neutron Irradiation of Supercritical Water

*66810*

*J. V. Zuffranieri*

## Abstract

In order to control the chemistry and the corrosion induced by nuclear power plants, we must understand many issues. One such issue is that of water radiolysis, or the decomposition of water caused by neutron, gamma, and beta radiation near the reactor core. The decomposition products formed by radiolysis can recombine to form corrosive species such as hydrogen peroxide and oxygen.

The aim of this project is to measure (G)-values of particular gases formed in high temperature water to determine radiolysis rates caused by neutrons, and to establish values for critical hydrogen concentration. The concentration prevents corrosive species from forming as a function of temperature up to and beyond the critical point. The impact of this research will be better selection of materials for current and future high temperature reactors, improvement of radiolysis assessment, improved water chemistry possibly leading to higher burnup fuel, the solution to supercritical water reactor design issues, and the promotion and maintaining of nuclear science infrastructure in the US.

## Accomplishments

We found that most of the very high temperature data (~385 °C and up at the irradiation site) for coolant water may have interferents due to the break down of water at these temperatures with wall reactions, etc. Since heat loss is high in the apparatus, we observed temperature drops of up to 200–300 °C between the main heater and the irradiation section. We found that the highest temperatures directly downstream from the main heater are limited to 570 °C for pure water and 450 °C for water with ethanol. We will install a new secondary cartridge heater to help us reach higher temperatures in the irradiation volume.

We performed deuterated alcohol and $N_2O$ experiments, where the reactions are the following:

$$N_2O + e^-_{(aq)} \rightarrow N_2 + O^-$$

$$H\bullet + C_2D_5OD \rightarrow HD + C_2D_5O\bullet$$

The HD data to the right of the pseudo-critical point (384.15 °C at 25 MPa) may not be valid since that is the sample temperature at which heater outlet temperatures get high enough to decompose ethanol.

We also performed critical hydrogen concentration (CHC) tests. We observed different results for different configurations. Before the lead section was put on, we detected a CHC of approximately 8E-5 molar hydrogen at PWR conditions. After adapting the experiment with the gamma shield, the CHC was harder to see, but we observed CHC behavior at PWR (2250 psi, 300 °C) conditions. Upon raising the temperature and pressure to supercritical levels, the CHC behavior seemed to at least dissipate. We need to run more tests to investigate the existence or non-existence of CHC in supercritical conditions.

# Molecular Simulation of Beta-Amyloid (A-beta) Peptide Interaction with Phospholipid Membranes

*69145*
*A. P. Thompson*

## Abstract

Our work focuses on the use of molecular simulation to probe the interaction of beta-amyloid (A-beta) peptides with phospholipid membranes. Recent experimental work of our collaborator (Theresa A. Good, University of Maryland) led to a hypothesis: that the binding of an oligomeric form of A-beta with cell membranes leads to neurotoxicity associated with Alzheimer's disease. However, fundamental information required for the development of effective treatments, such as the nature of the oligomeric species and the driving force for the peptide-membrane binding, remains unknown. We use molecular dynamics (MD) simulation with LAMMPS (Large-scale Atomic/Molecular Massively Parallel Simulator) as a means to gain this information. Since the time scales for MD (~10 ns) are not sufficient to observe entire peptide folding processes, we use the simulations in an iterative process that incorporates the experimental data of our collaborator.

## Accomplishments

In the past year, we constructed various atomistic models of A-beta peptide species (fragments and full monomers) in water. We also combined these A-beta models with a previously built atomistic model of a hydrated biological membrane composed of a bilayer of DPPC (dipalmitoyl phosphatidylcholine) molecules. We placed each A-beta molecule or fragment in various locations, in or near the membrane, and followed the molecular dynamics using LAMMPS. We implemented LAMMPS 2003 and 2004 for our parallel computing cluster comprising 100 dual processor Apple G5 server nodes. By following the dynamics for hundreds to thousands of picoseconds, we were able to reach some conclusions about (1) the stability of various conformations of the A-beta peptide in different regions of the membrane, (2) the effects of the peptide on the fluidity of the membrane, and (3) the role of electrostatics in the peptide-membrane interaction.

# Nanoscale Testing of Deformation and Fracture in Engineering Materials

*69146*

*N. R. Moody*

## Abstract

This is a joint research project between Sandia and Washington State University as part of the Department of Energy (DOE) Defense Programs Early Career Award for Scientists and Engineers awarded to Professor David F. Bahr. There are currently few experimental testing methods that can assess non-linear deformation and fracture phenomena on the µm and sub-µm length scales. Those that exist are often not verified using a second experimental approach. Nanoindentation techniques can probe failure modes, including film fracture and delamination, but these tests are difficult to verify without resorting to other indentation or scratching techniques. Therefore, we will apply a study that will allow for the development of testing using an independent technique (bulge testing and blister testing) to materials systems of interest to film substrate systems that can be used as models for components in the stockpile. We will perform additional testing in materials that exhibit non-linear deformation and shear band formation under high stresses. As both systems, nanoindentation and bulge testing, can be carried out in aqueous environments that are environmentally aggressive, we will perform testing correlation under various thermo-chemical environments to assure the robust nature of the experimental procedure. We will compare the results of all of these tests to simulations in projects supported in the Accelerated Strategic Computing Initiative (ASCI) program to show applicability of the testing methods to future verification of simulations for life cycle engineering.

## Accomplishments

The main tasks of this project focused on completing testing on the aluminum oxide-aluminum system. We unequivocally demonstrated that fracture occurs around the indenter during this testing. Secondly, we needed to demonstrate the addition of testing in aqueous solutions. Finally, we needed to demonstrate the applicability to comparing indentation testing to simulations.

We successfully identified circumferential cracking around indentations in anodic oxides of aluminum on aluminum substrates. These images are clearly proof that the resulting analytical fracture model for a hard film/soft substrate system is accurate. The fracture toughness ranges from 0.8 to 0.4 MPa–m. Secondly, we carried out in situ testing during electrochemical polarization of a stainless steel alloy in acidic chloride solutions at temperatures between 15 and 40 °C to determine the feasibility of performing these tests in harsh environments. Finally, we demonstrated the ability to utilize nanoindentation and atomic force microscopy to provide data that can be directly simulated in dislocation dynamics (DD) models. We demonstrated the correspondence between the slip behavior around an indentation and the DD simulation of that particular grain orientation in stainless steel.

## Refereed

K.R. Morasch and D.F. Bahr, “Nanomechanical Testing for Fracture of Oxide Films,” *Journal of Materials Research,* vol. 20, pp. 1490–1497, 2005.

M.J. Cordill, N.R. Moody, and D.F. Bahr, “The Effects of Plasticity on Adhesion of Hard Films on Ductile Interlayers,” *Acta Materialia,* vol 53, pp. 2555–2562.

A. Alamr, D.F. Bahr, and M. Jacroux, “Effects of Alloy and Solution Chemistry on the Fracture of Passive Films on Austenitic Stainless Steel,” to be published in *Corrosion Science.*

M.S. Kennedy, A.L. Olson, J.C. Raupp, N.R. Moody, and D.F. Bahr, “Coupling Bulge Testing and Nanoindentation to Characterize Materials Properties of Bulk Micromachined Structures,” *Microsystem Technologies,* vol. 11, pp. 298–302, 2005.

D.F. Bahr and G. Vasquez, “The Effects of Solid Solution Impurities on Dislocation Nucleation During Nanoindentation,” *Journal of Materials Research,* vol. 20, pp. 1947–1951, 2005.

## Other Communications

D.F. Bahr, K.R. Morasch, M.S. Kennedy, S.P. Anderson, A. Alamr, and N.R. Moody, “Through Thickness Fracture Behavior in Hard Films on Soft Substrates,”

presented at the TMS Annual Meeting, Mechanical Properties of Thin Films and MEMS Symposium, San Francisco, CA, February 2005.

# System Dynamics Modeling to Assist Regional Water Planning: Modeling the Non-Market Value of Water

*69156*

*V. C. Tidwell*

## Abstract

Fresh water is central to the vitality of our society; however, current demands are increasingly met through unsustainable practices. As such, improved management of our finite supplies is needed. Unfortunately, establishing such 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 by employing a cooperative modeling process formulated within a system dynamics decision framework.

Society values water in many different ways: economic, aesthetic, cultural, and instream/environmental. In some cases, these values can be directly quantified, as with economic valuation, while in others, quantifying its worth can be difficult. Nevertheless, these diverse value systems are playing an increasing role in establishing management plans that are acceptable, both publicly and politically. For this reason, we must develop and implement valuation methodologies within the water planning process.

We will pursue this work cooperatively with the University of Arizona and the National Science Foundation (NSF) Science and Technology Center for the Sustainability of semi-Arid Hydrology and Riparian Areas (SAHRA). SAHRA is a multi-institutional, multidisciplinary research program to improve understanding of the hydrologic cycle, to provide improved tools for decision makers, and to raise the level of hydrology literacy. By leveraging our efforts, we gain access to extensive expertise and data from unique studies on social valuation of water conducted in the Rio Grande and San Pedro river basins.

## Accomplishments

Through partnership between SAHRA, Sandia, and the US Army Corps of Engineers, we developed a survey instrument to address ongoing and planned bosque restoration in the Middle Rio Grande (MRG). We designed the survey to elicit new information about how the public views and values the MRG for inputs to a riparian valuation module. As river restoration is an established recipient of federal funding and is likely to increase in importance, the survey is also important as one of the first known economic valuation studies to focus on restoration. Most directly, the valuation of restoration allows improved cost-benefit analysis of water allocation in the MRG. Restoration research in the MRG is complemented by studies on the Salt and San Pedro rivers, similarly addressing the relationship between surface water, the environment it creates, and economic vitality.

We continue to research the non-market valuation of riparian services. This is a cutting-edge field with new academic and government studies frequently published. For the MRG, we included certain riparian services in the survey questionnaire, but others are beyond the scope of ordinary public awareness. A system dynamics framework to separate and enumerate these riparian services is in place to utilize value estimates as they become better established.

Recreational valuation of instream flows applied to the Aravaipa Canyon Wilderness in Southern Arizona is in press with the *Journal of Water Resources Planning and Management*, an American Society of Civil Engineers journal. Quantifying the value of recreational use of instream flow is an important component of the riparian valuation module within the generic watershed planning model.

## Refereed

M.A. Weber and R.P. Berrens, “Value of Instream Recreation in the Sonoran Desert,” to be published in *Journal of Water Resources Planning and Management.*

## Other Communications

M. Weber and G. Woodard, “Water Reuse Conservation Econometrics,” presented at the Arizona Hydrological Society Symposium, Flagstaff, AZ, September 2005.

M. Weber and S. Stewart, “Rio Grande Restoration Survey,” presented at the United States Society for Ecological Economics Conference, Tacoma, WA, July 2005.

# Interactive Water Quality Modeling to Assist Regional Water Planning

*69157*
*V. C. Tidwell*

## Abstract

While freshwater supplies are finite, demand has tripled since 1950. This growing disparity between supply and demand necessitates difficult decisions that often directly impact groundwater levels and stream flows. Perturbations to the 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 semi-arid 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 need to include biogeochemical processes 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. This work is in collaboration with the University of Arizona.

## Accomplishments

To improve our conceptual model of nutrient cycling, we acquired extensive data from the Rio Grande, which is representative of oversubscribed rivers in arid/semi-arid environments. Specifically, we sampled the 250 km reach between Cochiti Dam and Elephant Butte Reservoir in June, July, and August of 2004 to quantify the relationships between agricultural and urban land use, nutrient loading, and nutrient sinks within the surface water, hyporheic, and riparian systems. Data indicate that wastewater treatment plants are the largest and most consistent sources of inorganic nitrogen to the river. In both June and July, there was a net removal of nitrogen from the river as discharge decreased 26%, concentrations decreased 39%, and nitrogen loads decreased 56%. Interestingly, the diversion of river water for irrigated agriculture reduced discharge 25%, nitrogen loads 60%, and concentrations 47% along the same reach before draining back to the main stem of the river. This suggests that agricultural diversions were serving as a sink for nitrogen. However, in August, nitrogen loads were higher in returning drains, suggesting that agricultural systems had switched to a net source of nitrogen to the river. Possible reasons for this increase include higher rainfall in August, flushing accumulated solutes coupled with increased soil nutrients associated with the end of the growing season.

These findings imply that we must include both agricultural drains and wastewater treatment plants as point sources in the model. Furthermore, we must include these sources in a way that considers changes in behavior as a result of precipitation, vegetation, and hydrology. The stable isotope data also indicate the importance of understanding the hydrology of the stream to accurately model nutrient concentrations and cycling.

Subsequently, we quantified and implemented this conceptual nutrient cycling framework within a system dynamics context.

## Other Communications

G. Oelsner, P. Brooks, J. Hogan, H. Lacey, D. McDonnell, L. Zeglin, S. Mills, and J. Villinski, “Quantifying Nutrient Sources and Cycling Along the Upper Rio Grande,” presented at the 2005 Joint Assembly American Geophysical Union, North American Benthological Society, New Orleans, LA, May 2005.

G. Oelsner, P. Brooks, J. Hogan, H. Lacey, S. Mills, and J. Villinski, “Quantifying Nutrient Sources and Cycling

Along the Upper Rio Grande," presented at the 2nd International Symposium on Transboundary Water Management, Tucson, AZ, November 2004.

G. Oelsner, P. Brooks, J. Hogan, H. Lacey, D. McDonnell, S. Mills, and J. Villinski, "Quantifying Nutrient Sources and Cycling along the Upper Rio Grande," presented at the Geological Society of America Annual Meeting, Denver, CO, November 2004.

# Microbe-Exuded Polymers and the Enhanced Corrosion of Carbonate Materials

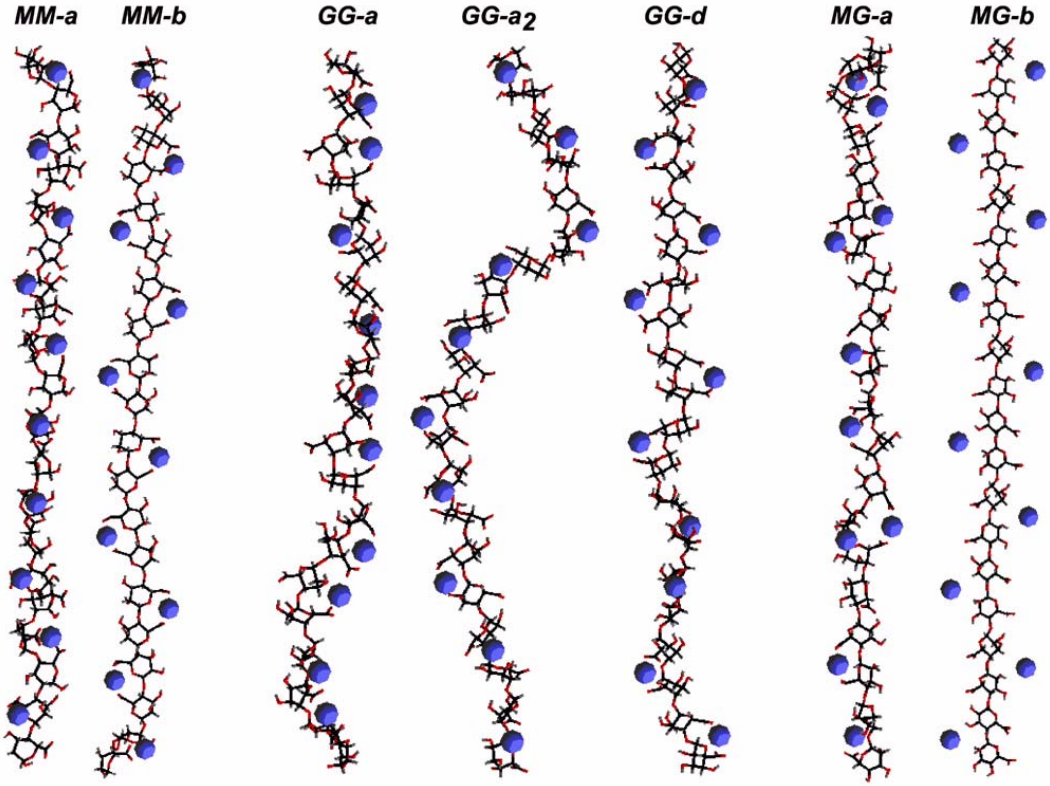
*69158*

*R. T. Cygan, M. S. Kent*


## Abstract

Cation binding by polysaccharides is observed in many environments. It is important for predictive environmental modeling and many industrial and food technology applications. The complexities of these organo-cationic interactions are well suited to predictive computer molecular modeling studies for investigating the roles of conformation and configuration of polysaccharides on cation binding. In this study, we chose alginic acid as a model environmental polysaccharide. We modeled representative disaccharide subunits and polymers, and we investigated the ability of subunits to bind calcium and extended these findings to modeling polymer interactions with calcium ions. The torsion angles (phi and psi) of the ether linkage between various monomeric subunits consistently have two energy-minima conformations for all representative dimer subunits, including mannuronic-mannuronic, guluronic-guluronic, and mannuronic-guluronic linkages. Cation binding by alginic acid has been attributed to the alpha 1-4 linkage between guluronic monosaccharides and the ability to form tetradentate complexes about cations.

Our theoretical studies indicate that some conformations of mannuronic-guluronic disaccharides have a higher binding energy than guluronic-guluronic disaccharides and may play an underdeveloped role in cation binding by alginic acid. We modeled subunits of alginic acid while interacting with the (104) surface of calcite. Constituent disaccharides of alginic acid have different binding capabilities with the mineral surface. Diguluronic acid forms the most stable complex by greater than 100 kcal/mol. The energy differences are related to the proximity and residence densities of the alginic acid electron donating moieties with calcium ions on the calcite surface. These are controlled, in


*Energy-minimized structures of alginate polymers in different torsional conformations corresponding to torsional energy minima.*

part, by the torsion of the ether linkage between monosaccharide units. The torsional conformation for the surface complex converts to the global potential energy minimum configuration when placed in aqueous solution, and is similar for all disaccharides (phi = 251.5 ± 34.7 °, psi = 234.7 ± 25.9 °) irrespective of the initial disaccharide conformation, which is similar to the previously achieved global energy minimum.

## Accomplishments

In terrestrial environments, polysaccharides are the second most abundant organic species following humic substances and can range from 5 to 30% of the total organic matter. Many of these polysaccharides are produced in microbial biofilms. These are thin films of microorganisms, including bacteria, archaea, cyanobacteria, algae, fungi, and lichens, growing on surfaces in an excreted layer of polysaccharide. The functionally diverse polysaccharide chemical structures vary by microorganism, growth stage, nutrient abundance, and other environmental stimuli. The three-dimensional structure of gelled biofilms is maintained by polysaccharides cross-linked by cations. The polysaccharides are generally composed of a variety of sugars and often contain functional groups (such as carboxylic acids) that can interact with the cations. The specific cation binding effects of polysaccharides depend on polysaccharide conformation and configuration, cation type, ligand functionality, acidic moieties, and pH.

This project applies the predictive capability of molecular modeling methods to explore the effects of conformation and configuration of the constituent dimers of a model polysaccharide on molecular stability, calcium binding, and polymer conformation. The ability to model and predict cation binding, as determined by polysaccharide structure, is desirable for a variety of industrial applications that require the dissolution of metal-bearing minerals; it is important for understanding cation binding in the environment and geochemical modeling. We selected alginic acid as a model environmental polysaccharide due to its well characterized chemistry and demonstrated presence in the environment. It is a straight-chain, hydrophilic, colloidal, polyuronic acid composed of guluronic (G) and mannuronic (M) acid residues configured in poly-G, poly-M, or alternating GM blocks. It is capable of chelating aqueous cations. Natural polysaccharides are produced from a variety of biological sources and exhibit chemical and functional diversity depending on the organism that produces them.

We used a detailed molecular level approach to understand the complex chemical contributions in conformation analysis and cation binding determinations. We used energy optimization and molecular dynamics methods to obtain chemical insights that may be difficult, if not impossible, to obtain experimentally. The use of these methods ultimately broadens the understanding of these interactions. We used the consistent valence force field (CVFF) to evaluate the potential energy of each conformation based on the torsional variation of the linking ether of the alginate dimer. We developed a computer algorithm to obtain a potential energy map based on the full exploration of the torsion phi-psi angles with resolution of 1 to 10 degree increments. We also used implicit solvation, employing a variable dielectric constant, to assess the impact of water on the conformation behavior. The energy values were three-dimensionally mapped to identify two to three unique energy minima configurations. Molecular dynamics simulations (NVT ensemble) for the various alginate dimers indicate the possible transitions and energy barriers among the conformers at room temperature conditions. We also completed several molecular dynamics simulations of the various water-solvated dimers interacting with a calcite cleavage surface. These calculations involve periodic simulation cells of approximately 5000 atoms.

We found the energy minima associated with the dimer conformations to have a global energy minimum for all disaccharides at phi = 274.1 ± 7.2 °, psi = 227.2 ± 4.8 °. We observed similar conformations to be most stable when the disaccharides were modeled with calcium ions and as larger (twenty unit) polysaccharide chains. We therefore predict that alginic acid in situ will commonly exist with this conformation. We modeled subunits of alginic acid while interacting with the (104) surface of calcite. Constituent disaccharides of alginic acid have different binding capabilities with the mineral surface. Diguluronic acid forms the most stable complex by greater than 100 kcal/mol. The energy differences are related to the proximity and residence densities of the alginic acid electron donating moieties with calcium ions on the calcite surface that are controlled, in part, by the torsion of the ether linkage between monosaccharide units.

## Refereed

T.D. Perry, R.T. Cygan, and R. Mitchell, “Molecular Models of Alginic Acid Including Interactions with Calcium Cations and the (104) Surface of Calcite,” submitted to *Geochimica et Cosmochimica Acta*.

T.D. Perry, R.T. Cygan, and R. Mitchell, “Molecular Mechanics Force Field for Modeling Carbonate Mineral Surfaces with Explicit Hydration,” submitted to *Langmuir.*

# Adaptive Algorithms for Use in the Rejection of Periodic Disturbances of Unknown Frequency

*69166*
*E. S. Hertel Jr.*

## Abstract

This research will involve the development of new adaptive algorithms for use in the rejection of periodic disturbances of unknown frequency. The particular area of application involves problems encountered in active noise cancellation and vibration control. The topics explored involve the application of adaptive cancellation algorithms to systems with resonant modes, as well as the pursuit of more globalized cancellation. This involves the use of multi-input, multi-output adaptive disturbance rejection algorithms. Exploration of various higher order input/output configurations will build upon research started at the University of Utah. Also, the cancellation of disturbances consisting of rapidly time-varying frequency will be examined.

## Accomplishments

We are continuing to develop new algorithms for the rejection of periodic disturbances 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. We considered algorithms that identify and adapt to variations in either disturbance or plant parameters. By assuming steady state responses, we derived simple and robust algorithms with established convergence properties.

The exploration of algorithms that assume known plant dynamics and unknown disturbance characteristics builds on work previously conducted at the University of Utah. Using an offline estimate of the plant frequency response, gradient based techniques achieved as much as 97% attenuation of a sinusoidal disturbance of unknown frequency through multi-channel active noise control (ANC) experiments. We performed comparison of higher order square and non-square plant configurations.

Microphones being cheaper than speakers, it is typical to use more error sensors than to control sources. Therefore, we will continue to examine the effect of adding additional sensors to multi-channel configurations.

The algorithm utilizes an indirect approach in which we first obtain an estimate of the frequency and then use it an algorithm for known frequency. A crucial component of this method for the rejection of periodic disturbances of unknown frequency is frequency estimation. We showed that a phase-locked loop approach to online frequency estimation has been shown to work in conjunction with the disturbance rejection algorithms through both single-channel and multi-channel ANC experiments. We also demonstrated the ability to track time varying frequencies with a mean tracking error of 0.68 Hz while still achieving significant disturbance rejection. The thorough investigation of unknown sinusoidal disturbances acting on known systems laid the foundation for the exploration of disturbances acting on unknown systems.

Since many applications involve unknown and time varying dynamics due to variations in environmental factors, we developed methods for identifying plant parameters online. We developed a new algorithm that uses a least-squares technique to continuously update an estimate of plant dynamics. We then used this estimate in determining an appropriate control signal. Based on a set of linear equations describing the plant output over time, a recursive least-squares algorithm with exponential data forgetting and stabilized covariance matrix achieved 97% attenuation of a sinusoidal disturbance through single-channel ANC experiments. Conditions for exponential convergence have been satisfied. Also, we verified the ability to track time varying system parameters and maintain considerable disturbance rejection. Preliminary results established the feasibility of merging components of the various algorithms considered to reject sinusoidal disturbances of unknown frequency acting on unknown systems.

# Explicit and Implicit Measures in Human Aversive Classical Conditioning

*74759*
*M. C. Grubelich*

## Abstract

Classical conditioning provides a proven and accessible way to study the differences and interactions between explicit and implicit knowledge. Using physiological measures (such as skin conductance) as an index of implicit factors, our work with the California Institute of Technology in the past year demonstrated a dissociation in the neuronal representation of explicit and implicit systems. We also know about localized structures in the brain that appear to mediate interactions between the two systems. These findings provide evidence that the neuronal representations of explicit and implicit knowledge (as well as their interaction) can be tracked using functional Magnetic Resonance Imaging (fMRI) and physiological measures.

In FY 2005, we studied the influence of cognitive distractions on implicit processes. We proposed using dynamic causal modeling to study the differences in functional connectivity in the brain that occur as a result of the interaction of explicit and implicit knowledge during conditioning. Dynamic causal modeling is a forward neuronal model that is capable of describing the effects one area of the brain has on another. Analysis of conditioned associations using dynamic causal modeling would allow us to better understand the interactions between explicit and implicit brain systems. Knowledge of the timing and nature of implicit influences on explicit decision making provides a direct means of understanding when and how decisions are being made that are counter to equilibrium predictions. It also provides a means for understanding how a subject's perceptions of a situation are shaped during the acquisition of implicit factors.

Understanding the discrepancy between the optimal decision and reality then depends on understanding the differences between implicit and explicit factors. Any hope to influence or optimize group decision making pivots on the interaction between explicit (rationally describable) and implicit (intuitive) factors.

## Accomplishments

- *Experiment Completed:* CalTech conditioned 12 subjects to two classes of stimuli, faces and abstract figures, while recording brain activity using fMRI. The abstract figures were the same used in experiments during our first year.
- *Results:* Initially, we confirmed that there are indeed differences in the neuronal representations of abstract figures and faces. Abstract figures tend to be better represented in mid-level visual areas that surround the primary visual cortex, called extra-striate cortex. The presentation of faces elicits greater responses throughout most of the brain, including the area of the brain that represents fear, the amygdala, and an area known to represent faces, the fusiform face area.

In order to look at conditioning related changes in the brain, we tested for regions where activity correlated with associative value. Surprisingly, significant portions of the brain correlate with the associative value of the stimulus. These areas include the first few steps of visual processing in the thalamus. Since it is difficult to imagine these early areas differentially representing two faces (or two abstract figures), it is more likely that these thalamic areas are driven by inputs from higher visual areas and possibly areas involved in aversive learning such as the amygdala or insula.

We also examined times when the brain's expectations differed from actual events, called prediction errors. Areas of the brain that reflected prediction errors included portions of visual cortex and another thalamic nucleus. Interestingly, an area considered to be the serotonin center of the brain (dorsal Raphe) also correlates heavily with prediction error. This is consistent with the hypothesis that serotonin serves as a teaching signal for aversive learning.

# Maximally Autonomous Autodirective Antenna Array Technology

*75786*

*R. A. Bates*

## Abstract

Antennas are the critical link between the relatively controlled domain of radio frequency (RF) guided waves and the radiated propagation environment, passively filtering signal energy with respect to operating frequency, polarization state, and direction. All undesired signals are noise to a receiving system and directly reduce system performance. Simply directing the antenna toward a desired source, and away from interference sources, results in significant improvements in link quality by enhancing the signal to noise ratio (SNR) at the receiver. The effective bandwidth available to all users in a shared environment also increases.

We will explore technology that addresses the significant and high value goal of implementing a class of practical antenna solutions that use automatic electrical array pointing with minimal loading on the original target system. Target tracking of aircraft or vehicles in intelligence applications requires quantitative understanding of electrical switching latency times. Antenna beam steering properties must be subject to network algorithm control.

Applications for steered beam antennas exist in intelligence ground sensor applications and reentry vehicle (RV) flight testing. RV communications with stationary communications satellites require dynamic control of the antenna beam. We focused on mission areas where limitations exist on emitter power, space, and cost, such as unmanned aerial vehicles (UAVs) and exoatmospheric communications systems, nano-satellite constellations, distributed remote sensing networks, and personal communications. Many of these applications demand more performance improvement than can be offered through enhancements in other, more exhaustively addressed areas of RF communications research, such as error coding, modulation techniques, or amplifier efficiency improvement. The primary end goal of the research effort is the evaluation of low overhead beamforming techniques applied to small antenna arrays to implement a maximally autonomous autodirective antenna function. This work is in conjunction with New Mexico State University.

## Accomplishments

The technical accomplishments are primarily in the area of analyzing, simulating, and making measurements with near earth and subsurface RF communications. This included analyzing surface mode traveling waves along the earth-air interface. It also included analytical performance estimates for vehicle mounted conformal arrays. We performed and analyzed numerous computational simulations modeling a specific system intended to excite bound subsurface wave propagation at the earth-air interface. Limited measurements using buried RF couplers were also performed. We delivered these results as a formal report at the end of the spring semester. They show the potential for overcoming non-ideal terrain in diffraction limited frequency bands, establishing links over non-line of sight channels, and decreasing the probability of intercept for near-earth located RF links. A key finding of the work is that antennas operating near earth or in the earth are more appropriately treated as probes attempting to excite a dielectric waveguide, as opposed to radiating elements operating in a homogeneous medium. This finding changes several aspects of system design when attempting to use other than free-space propagating modes for communications in such environments.

We performed additional research in the areas of adaptive antenna elements and RF control circuits, in line with the intended future work of the fellowship recipient. Literature searches reveal that the intended body of work is, as a whole, relatively unexplored and thus worthy of pursuit. Making initial application of the intended auto-adaptive techniques to near earth propagation environments will serve to tie the previous work with the more degree specific focus of the future efforts of the student.

# Generalized Continuum Models for Inelasticity in Solids: Formulation of Theories, and Variational Methods for Computation

*78783*

*T. D. Nguyen, K. R. Garikipati*

## Abstract

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. These theories are capable of modeling the influences of microstructure and have gained prominence over the past decade. They are applied 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 order 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 (1) the order of strain gradients, (2) the role of higher order derivatives in governing equations, (3) inelastic strain gradient dependence in yield functions and flow rules, and (4) the application of additional boundary conditions.

In this project, we propose to work with a class of generalized continuum inelasticity models. 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. In particular, we will draw heavily from dislocation theory. We propose to develop vector finite elements and discontinuous Galerkin methods to solve initial and boundary value problems using these models.

## Accomplishments

We successfully implemented a strain gradient plasticity formulation incorporating the Bammann-Chiesa-Johnson (BCJ) model using vector finite element methods. The motivation for this choice is that the plastic strain gradient tensor, defined as the curl of the plastic deformation gradient, is similar in form to the vector fields obtained in electromagnetism for which application vector finite element methods were developed. This formulation was developed in the context of anti-plane shear problems where this plastic strain gradient effect is pronounced. The formulation performs well in comparison with standard finite element implementations. In general, it is stable for a wider range of interpolation combinations for displacement and plastic strains. A student obtained his doctoral degree in August 2005 working on this problem, and a journal publication is in preparation. Separately, we developed a family of discontinuous Galerkin finite element methods for strain gradient damage problems. The aim of this formulation is to enable the use of low order (C^0) interpolations for strain gradient problems in which the governing equations are higher order nodes (typically, fourth order). The alternative is to use mixed methods for such problems, introducing the requirement to satisfy the inf-sup condition. We completed convergence studies and an error analysis for this class of discontinuous Galerkin methods, which will now be extended to strain gradient plasticity models.

## Refereed

G.N. Wells, K. Garikipati, and L. Molari, “A Discontinuous Galerkin Formulation for a Strain Gradient-Dependent Damage Model,” *Computer Methods in Applied Mechanics and Engineering*, vol. 193, pp. 3633–3645, 2004.

L. Molari, G.N. Wells, K. Garikipati, and F. Ubertini, “A Discontinuous Galerkin Method for Strain Gradient-Dependent Damage: Study of Interpolations and Convergence,” to be published in *Computer Methods in Applied Mechanics and Engineering.*

# 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*

## Abstract

Modeling failure in geomaterials due to various loading and environmental conditions is a challenging problem that 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 fragmented geologic material. As one approach to modeling this transition, we propose to develop physically based cohesive zone models for geomaterials and their numerical implementation via the finite element method. This would be one tool in developing a complete, predictive computational modeling program for failure analysis of geomaterials. This work is in collaboration with Stanford University.

Two problems that would be better understood with such a modeling capability are the defeat of Hard and Deeply Buried Targets (HDBTs) 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 1000s of years) of deep geologic nuclear waste repositories. Obtaining Nuclear Regulatory Commission (NRC) approval becomes difficult if the Department of Energy is restricted to using existing methodologies. In addition to modeling the defeat of HDBTs 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 over consolidated clay encountered in tunneling construction, oil and natural gas production, and depleted reservoirs used for subsurface sequestration of greenhouse gases.

## Accomplishments

During the past year, we formulated a rigid plastic cohesive zone model for geomaterials and implemented the model in an embedded strong discontinuity finite element. We demonstrated this embedded strong discontinuity technique in previous publications to yield solutions that are insensitive to mesh refinement and element alignment. The constitutive law on the fractured surfaces is based on that discussed in the literature. This model captures many of the phenomena observed in the frictional sliding of geomaterials, including stable and unstable slip along surfaces, and dependence of the friction angle on slip speed and state of the surface contacts. For improved accuracy and stability, we used a generalized trapezoidal scheme to implement the constitutive model.

Several aspects of our model's behavior need to be addressed in a more robust fashion to accurately capture the localization of these materials. We need to model the transition of the material from intact continuum to a body with a fractured surface in a more physically based, rather than phenomenological, manner. We will assess the softening behavior exhibited using a mode II fracture energy, as discussed in the literature. We will also investigate and determine the optimal implementation of the algorithm that tracks the propagation of the surface through the mesh. While the implementation of the element is compatible with many bulk constitutive models, including von Mises, Drucker-Prager, and the Sandia Geomodel, we performed very little testing against experimental data. We plan to perform parameter fitting and validation using experimental data for both the San Marcos gabbro and Fichtelbirge granite.

## Refereed

C.D. Foster, R.A. Regueiro, A.F. Fossum, R.I. Borja, "Implicit Numerical Integration of a Three-Invariant, Isotropic/Kinematic Hardening Cap Plasticity Model for Geomaterials," to be published in *Computer Methods in Applied Mechanics and Engineering.*

# Reconciling System and Application Logs

*80590*
*N. A. Durgin*

## Abstract

Computer system event logging is best done as a fine balance between overflow of information and lack of data. Limited logging can hinder both 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 human capital necessary to examine the logs for pertinent data is exorbitant. 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 that 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 determining by hand which high layer event corresponds to which low level entry. Such aggregation is surprisingly complex in today's modern computing environment.

The goal of this research is to devise a method to automate the process of mapping events across system layers in order to assemble them into useful models of individual events. Such models will aid in both forensic analysis of compromised machines and intrusion detection systems. They will provide the templates necessary to create useful, easily analyzable logs. This work is in collaboration with the University of California at Davis.

## Accomplishments

This year's research focused on developing scripts to automate the process of aggregating various system logs and extracting the log data into a more readable format to simplify the log analysis task for system administrators. The aggregating script gathers entries from input logs (either multiple logs from the same system, or logs from across multiple systems) that match a given search pattern, and then arranges the matching log entries into a single chronological log file for analysis.

The extraction script is designed to use pattern matching across the log file to detect pre-defined templates of log entries ("footprints") and to replace each instance of a given footprint with a single line describing, in a more easily accessible format, the footprint detected. For example, the string of lines in a strace log file that describes the dynamic load of a shared object file, often numbering at least six lines of bare system call events, can be replaced with a single line telling the system administrator, "Dynamic log of shared object file /etc/foo.so.1."

We honed the aggregation script to work on Red Hat system logs, and it is presently capable of detecting log entries matching the desired string, and aggregating those entries into a single chronological log file.

The extraction script developed to work on strace log files is more in a proof-of-concept stage, as the process of developing footprints proved more difficult than originally expected. At present, it is capable of reducing overall log length by an average of 35% and replacing an average of 72% of log file entries (system call traces) with more accessible descriptions of the system trace.

# Collaborative Distributed Transceiver Designs and Protocols for the Interface of Wireless Sensor Networks and Wireline Infrastructures

*80591*
*N. M. Berry*

## Abstract

The collaboration between wired and wireless networks represents a unique opportunity to expand techniques traditionally used in Internet-based infrastructures to a wireless networking domain. This research focused on the development of a mobile agent based framework designed to run on resource limited wireless sensor node technology. The lack of bandwidth, memory, and power constrains the agents to sizes of 20–200 bytes to support limited communication of agents with attached data. The framework exposes the trade-off between communication reliability and overhead. The principle is that algorithms should be designed to tolerate lossy communications. The first year of work provided a coherent view of how technology like agent based systems can be adapted to a traditionally resource restricted environment.

## Accomplishments

In the past year, our research focused on the development of a mobile agent based framework designed to run on resource limited wireless sensor node technology. The design is driven by tight constraints on bandwidth, memory, power, and reliability. In our context, we defined agents as small bytecode programs (generally 20–200 bytes) that execute in a protected environment. We call the agents "mobile" because agent state (memory) is maintained when agents move from one node to another. Propagation can be self-directed via unicast, broadcast, or multicast. This approach is in contrast to the traditional approach, which we term "monolithic" programming: the development of one program that is distributed to all nodes in the network. Agents, on the other hand, are tailored to specific duties, enabling more functionality in the network and/or a reduction in overhead to deploy specific behaviors in the network. As with a monolithic program, the behavior of the system as a whole is formed by the interactions between its constituent nodes. The advantage to the agent approach is that the various behaviors are expressed succinctly and separately as individual agents.

Our agents communicate using an efficient and localized primitive agent inspired by biological pheromones. Our pheromones are defined by a scent ID, a source ID, and a strength. When a node deposits a pheromone with a given strength, the information propagates across a limited geographic space and decays over time if not reinforced. This method ensures that stale data will expire, encourages localized communication, and is tolerant of dynamic/sporadic connection topologies.

The first year research encompasses the following technical accomplishments: We designed and implemented an agent framework that runs on any TinyOS-compatible sensor node. The framework leverages the Maté virtual machine and adds agent mobility and communication aspects. The framework is implemented in network sensor C (nesC), but user agents are coded in a high level scripting language that is part of Maté, called TinyScript.

We can simulate agent scenarios using the TinyOS SIMulator (TOSSIM). In this way, we can write and test code prior to deployment on physical nodes, and we can perform simulations of large-scale deployments. An example scenario explored is a tracking application in which agents are distributed to the perimeter of a sensor network. Any event that crosses the perimeter is tracked by a "swarm" of agents. This application demonstrates in-network processing, scalable node density, and efficiency benefits of the mobile agent approach. We demonstrated the agent framework on 25 Moteiv Telos nodes that run TinyOS.

We developed a simulator to improve exploration of the problem space, framework services, and scalability. Our agent's high level pythonic simulator (AHLPS) scales to much larger simulations than TOSSIM and considerably eases the burden of writing and testing new algorithms. These benefits are enabled by a simpler radio model and the use of the Python language to implement framework services. Once an algorithm has been validated under AHLPS, it can be implemented in nesC and tested more extensively using simulation and physical experimentation.

The results of our first year research indicate that the agent programming approach can be more efficient than the monolithic approach for some applications and, in general, can be at least as efficient in most applications (through additional development of the framework, such as the caching of agent code). The pheromone communication primitives show great promise, and we will continue to develop it in year two.

## Other Communications

L. Szumel, "A Sensor Network for the Study of Invasive Species Growth," presented at the John Muir Institute of the Environment Open House, Davis, CA, November 2004.

L. Szumel, J. LeBrun, and J.D. Owens, "A Framework for Sensor Network Mobile Agents," presented at the CITRIS Corporate Sponsor Day, Davis, CA, October 2004.

L. Szumel, J. LeBrun, and J.D. Owens, "Towards a Mobile Agent Framework for Sensor Networks," presented and in *Proceedings of the Second Workshop on Embedded Networks*, Sydney, Australia, April 2005, vol. 2, pp. 79–87.

L. Szumel, "A Mobile Agent Framework for Sensor Networks," presented at the Tiny OS Technology Exchange, Berkeley, CA, March 2005.

L. Szumel, "A Novel Pheromone Approach to Spatial Clustering," presented at the Second Annual IEEE SECON, Santa Clara, CA, September 2005.

# Ultrafast Low Voltage MEMS Switches for Optics and RF Applications

*80592*
*G. N. Nielson*

## Abstract

A new actuation technique for microelectromechanical systems (MEMS) switching is proposed that will provide switching times of 100 ns, require actuation voltages of less than 10 V, and provide a switch displacement of at least 1 µm. This technique makes use of stored elastic potential energy to drive the switching motion while using electrostatic attraction to hold the switch in place between switch operations. The energy used for switching is largely retained within the system rather than being dissipated. This allows the energy to be recycled and used repeatedly for switching, resulting in very low energy consumption. These performance characteristics represent a dramatic improvement over all other MEMS switch actuation methods.

The switching technique can be used to create vertical or horizontal rectilinear motion, or torsional motion. This flexibility allows the switching technique to be used for both free space and integrated (waveguided) optical switches and radio frequency (RF) MEMS. As part of the proposed work, we will demonstrate both RF and optical switch prototypes. RF and optical MEMS switching are widely explored due to the potential impact in both defense and commercial applications; however, there are a number of performance challenges to be overcome before devices can be successfully deployed. By utilizing the proposed switching technique for RF and optical switching, most, if not all, of the performance issues hindering the development of these switches can be addressed. PI Gregory Nielson is a recipient of the post-doctoral Truman Fellowship Award.

## Accomplishments

We achieved a number of significant accomplishments. These accomplishments were in the areas of design and fabrication of devices, demonstration of the proposed switching concepts, and building up the infrastructure necessary for the testing of devices related to the proposed work.

In the area of experimental demonstration, we obtained two particularly important results. First, we demonstrated MEMS switching by using stored elastic potential energy as a source of switching energy for the first time. This provided switching at speeds between 350 to 500 ns over a displacement of 1.8 µm using only 35 V. This is the fastest switching speed reported for devices with a comparable displacement and voltage by roughly an order of magnitude. A second experimental result demonstrated resonant MEMS pull-in (for switch initialization) for the first time. This allowed the pull-in of an electrostatic MEMS device at a voltage level that was 25% less than the pull-in voltage (the voltage that is typically required for pull-in).

*Researcher Gregory Nielson is working to develop a new actuation switching technique for MEMS that can be used to create vertical or horizontal rectilinear motion or torsional motion.*

There were many accomplishments in the area of design and fabrication. We designed a number of devices. The first devices designed were an array of MEMS switching structures with switching speeds ranging from 0.5 to 25 MHz. We fabricated these devices and used them to demonstrate the switching concepts proposed. The devices will continue to serve as a platform to optimize switching control.

Additional devices designed are torsional and horizontal motion RF MEMS switch structures that use the new actuation technique. We will fabricate these first prototypes using Sandia's Au (gold) RF MEMS switch process. In addition, we designed some MEMS torsional mirrors for high speed actuation (sub-microsecond).

We accomplished much work related to the infrastructure of this research for both design and testing. We developed a multi-physics finite element model that predicts the pull-in voltage and associated stress and strain of the test devices, allowing better design approaches and optimization as well as offering better understanding of the experimental performance of the devices.

To facilitate testing of the devices, we designed and assembled a vacuum chamber test setup that included adjustable mounts for packaged devices, electrical feed throughs, and a window for device monitoring. This proved to be a crucial piece of equipment for the testing of devices. Also, to better control the switching, we conducted a preliminary evaluation of open-loop and closed-loop approaches for resonant pull-in for device initialization.

Seven papers related to this work have been accepted for publication in peer-reviewed journals and conference proceedings. One additional paper is currently in review. We submitted five technical advances related to this work. We will submit several additional technical advances soon.

## Refereed

D. Seneviratne, G.N. Nielson, S. Takahashi, G. Barbastathis, and H.L. Tuller, "On the Use of Titanium Nitride as Structural Material for Nano-Electromechanical Systems (NEMS)," presented and in *Proceedings of the IEEE Conference on Nanotechnology,* Nagoya, Japan, July 2005, vol. 1, pp. 4–5.

S. Takahashi, G.N. Nielson, D. Seneviratne, G. Barbastathis, and H.L. Tuller, "Titanium Nitride Micro-Electromechanical Bridge for Wavelength Selective Switching," presented and in *Proceedings of the IEEE/LEOS International Conference on Optical MEMS and Their Applications,* Oulu, Finland, August 2005, vol. 1, pp. 41–42.

G.N. Nielson and G. Barbastathis, "A Physically Based Model for Dielectric Charging in an Integrated Optical MEMS Wavelength Selective Switch," presented and in *Proceedings of the IEEE/LEOS International Conference on Optical MEMS and Their Applications,* Oulu, Finland, August 2005, vol. 1, pp. 69–70.

G.N. Nielson, D. Seneviratne, F. Lopez-Royo, P.T. Rakich, Y. Avrahami, M.R. Watts, H.A. Haus, H.L. Tuller, and G. Barbasthatis, "Integrated Wavelength-Selective Optical MEMS Switching Using Ring Resonator Filters," *IEEE Photonics Technology Letters,* vol. 17, pp. 1190–1192, June 2005.

L. Waller, S. Takahashi, G.N. Nielson, D. Seneviratne, H.L. Tuller, and G. Barbastathis, "Microelectromechanical Wavelength-Selective Switching for Integrated Optics," to be published in *2005 ASME International Mechanical Engineering Congress and Exposition.*

G. Barbastathis, G.N. Nielson, C. Wong, "Three-Dimensional Optics," to be published in the *12th International Display Workshop.*

G.N. Nielson and G. Barbastathis, "Dynamic Pull-In of Parallel Plate and Torsional Electrostatic MEMS Actuators," to be published in *Journal of Microelectromechanical Systems.*

# Catalyst Development and Micro Reformers for Fuel Processing

*80593*
*D. Ingersoll*

## Abstract

Micro fuel cells developed at Sandia and elsewhere are able to generate sufficient power for a variety of electronic devices, such as micro robots, radio frequency tags, and distributed microsensor systems. However, to generate this electrical energy, a ready source of hydrogen fuel is required. Several strategies are being developed to meet these fuel needs. In this project, we propose to develop a micro reformer for the conversion of readily available biomass-derived hydrocarbons into hydrogen and integrate this device with a small fuel cell.

This task will focus on design, development, fabrication, and finally the evaluation of the micro fuel processor used for the reformation of a carbohydrate feedstock, specifically glucose, to hydrogen. The proposed micro-fuel processor is based on a bench top scale glucose steam reforming system built using commercially available hardware. The use of micro-channel reactors will demonstrate (1) the feasibility of fuel processing on the microscale, and (2) carbohydrate micro reforming.

In addition, the possibility for large scale production of hydrogen based on the micro-reformer exists, thereby helping to realize the US security strategy for energy self-sufficiency. The approach in this case would not be to merely scale-up the reformer in size, but instead to have a large number of micro-reformers operating in parallel to one another. This "numbering-up" approach for large scale hydrogen production offers some interesting possibilities, such as the ability to precisely and easily tailor the hydrogen output need while maintaining high system efficiency.

Initial reforming experiments will utilize commercially available Pt catalyst supported on -325 mesh $Al_2O_3$. Deposition and adhesion tests are ongoing for placement of the catalyst in the reactor channels. We also plan to utilize novel catalytic membranes of Pt supported on NaY zeolite that we previously developed for handling liquid streams. This work is being performed at New Mexico Tech.

## Accomplishments

The micro fuel processor is modeled on a bench top scale glucose steam reforming system. We built it using commercially available microreactors, parts, and catalysts. The use of a microchannel reactor will demonstrate the feasibility of fuel processing on the microscale. In addition to size and mass advantages, micro reactors have the additional advantage over large scale reactors of decreased surface:volume ratios and increased thermal conduction and mass transfer rates. Steam reforming has the highest theoretical efficiency and potential $H_2$ selectivity compared with other reforming methods; and biomass is a desirable fuel, as it is abundant, renewable, and carbon cycle neutral. We suggest one of the major and most easily extractable components of biomass, glucose, as a test fuel.

We completed the pressurized section of the experimental apparatus. This section of the fuel reforming system consists of a Hitachi L-6000 high performance liquid chromatography (HPLC) pump, a gas phase microreactor with mixer and internal heating from the Institute of Molecular Manufacturing, pressure and temperature sensors upstream and downstream from the reactor, a heat exchanger, a 500 cc flash tank, and a 150 cc collection cylinder for liquid products. The HPLC pump feeds the aqueous fuel stream to the reactor at controllable pressures. The microreactor is mounted on an optical breadboard for stability. The flash tank is used for condensation of gaseous hydrocarbons and separation of the gas and liquid phases. We installed pressure and temperature sensors upstream and downstream from the reactor. The heat exchanger consists of an ~14 cm length of 316 stainless steel tubing surrounded by a PVC water jacket with a high temperature epoxy binding the tube to the PVC jacket.

We pressurized and leak tested all components of the system to 10 bars using distilled $H_2O$. A volume analysis of the collection section (from the bleed valve above the flask tank to the collection cylinder) is necessary for performing a mass balance to determine quantities of gaseous and liquid products produced in the microreactor.

After leak checking, we used an air purge to depressurize the system, to extract the $H_2O$, and to

locate possible fluid stagnation points other than the collection tank. While most of the water drained into the collection tank, water stagnated in the flash tank and cross tee. We are considering design modifications for the collection stage to eliminate liquid trapping in the nearby tees and valves. The replacement of the bellows valve below the flash tank with a bleed valve will eliminate the bottleneck responsible for water accumulation in the flash tank. We will use nitrogen to purge the reactor system during routine operation. When fully operational, the flash tank will contain gas and water condensate on the walls with no standing liquid at the base; all liquid phase reactor effluent will accumulate in the collection tank.

We obtained a quadrupole mass spectrometer to measure gas composition. Initial experiments will use a Pt catalyst supported on -325 mesh $Al_2O_3$. We are conducting deposition and adhesion tests for placement of the catalyst in the reactor channels. Future work includes system calibration and optimization of temperature, pressure, flow rate, and catalyst loading in the reactor system for hydrogen selectivity.

## Other Communications

K. Norman, V. McIntire, and D. Hirschfeld, “Fuel Cost Analysis for Reforming of Biomass-Derived Hydrocarbons,” presented at the 206$^{th}$ meeting of the Electrochemical Society, Honolulu, HI, October 2004.

# Catalytic Membrane Development for Microscale Glucose Reforming

*80594*
*D. Ingersoll*

## Abstract

The objective of this project is to modify our recently developed transition metal/zeolite catalytic membranes for conversion of biomass derived hydrocarbons to hydrogen through liquid-phase reaction. We prepared both Pt/NaY and Pt-alumina catalysts and studied them for reformation of both methanol and glucose. The catalytic membrane is expected (1) to enhance the yield by the improved dispersion of metal species in the zeolitic channels and the synergistic effects of metal-zeolite on the dehydrogenation reaction, and (2) to increase the $H_2$ selectivity by reducing the residence time of the produced $H_2$ on the catalyst surface and in the solution phase that, in turn, minimizes the $H_2$-consuming side reactions.

During operation, the reaction zone is a Pt/NaY thin membrane layer (3–5 µm thick). The pressurized feed solution transports through the sub-nanometer metal loaded zeolite pores where the oxygenated hydrocarbon molecules are catalyzed and converted to $H_2$ and $CO_2$. The produced $H_2$ and $CO_2$ will be released instantly to the vapor phase on the other side of the membrane where a lower pressure is maintained. While both catalysts exhibit good behavior at the reaction temperatures studied (220 and 260 °C), the Pt/NaY catalysts performed better than Pt-alumina, even when the Pt/NaY catalysts had much lower Pt loadings. Preliminary data also show that for both catalysts, the $H_2$ productivity was significantly enhanced by increasing the reaction temperature from 220 °C to 260 °C. Based on the current results, we identified the specific conditions for Pt/NaY catalytic membrane synthesis and reaction operation. This work will be performed at New Mexico Tech.

## Accomplishments

During the first year of the project, we synthesized and evaluated Pt-alumina nano-catalysts and Pt/NaY particulate catalysts having different Pt loading levels for the conversion of liquid methanol feedstocks.

We accomplished the synthesis of nanodispersed Pt-alumina catalyst using a sol-gel approach, and the Pt catalyst was loaded by an incipient wetness impregnation technique followed by drying, calcination, and reduction in $H_2$ flow at 260 °C. With this approach, we observed no distinguishable PtO and/or Pt peaks in the x-ray diffraction (XRD) due to the extremely high dispersion state and small size of the catalyst. We completed scanning tunneling electron microscopy (STEM) of the samples, and these clearly indicate uniform dispersion of the Pt particles on the alumina nanoparticle surface and having uniform Pt particle size distribution of around 2 nm. We used the STEM/Energy Dispersive Spectrometer data in conjunction with microprobe analysis to quantify the level of Pt loading in various samples prepared using this approach, and found these to be 0.5%, 1.1%, and 2.9%.

We accomplished synthesis of Pt/NaY particulate catalysts by first preparing NaY zeolite particles by hydrothermal synthesis, and then loading the Pt into the zeolite by ion exchange using a dilute solution of $[Pt(NH_3)_4](NO_3)^{-2}$ at 80 °C under reflux. The sample was then washed, dried, calcined, and subsequently reduced in a $H_2$ flow at 260 °C. We examined the crystal structure of the zeolites and catalysts by XRD, and we determined the Pt loading level of the Pt/NaY by microprobe analysis. We prepared Pt/NaY catalysts with Pt loads of 0.2% and 0.4% for further evaluation.

We evaluated the Pt-alumina catalysts in a packed-bed reactor for the conversion of $CH_3OH$ to $H_2$, and we conducted these experiments at 220 °C (380 psi) and 260 °C (760 psi). In all cases, we fed a 1 wt.% methanol solution in water into the reactor at a flow rate of 0.8 g/h. We continuously monitored the $H_2$ production rate during 22 hours of operation. In every case, we used 1g of each catalyst (0.5 wt%, 1 wt% and 3 wt% $PtA1_2O_3$ and 0.2 wt% Pt/NaY) in the tests. We observed that hydrogen production rates slowly increased during the first 400 minutes of the evaluation and thereafter ran at a fairly constant production rate for the duration of the test. Production rates achieved in these tests varied between $5x10^{-6}$ and $8x10^{-6}$ mol g $min^{-1}$ depending on the catalyst used. We also observed that the Pt/NaY catalysts performed better than the Pt-alumina catalysts, even when the Pt/NaY catalysts had Pt loadings much lower than those of the Pt-alumina catalysts. Preliminary data also show that we significantly enhanced the $H_2$ production rate by increasing

the reaction temperature from 220 °C to 260 °C for both the Pt/NaY and $PtAl_2O_3$. The conversion rates and selectivity observed are comparable to the best values so far reported. In some cases, higher conversion rates reported in the literature may in fact by due to higher fuel flow rate. Additional work to further explore these conversion efficiencies and to prepare and evaluate other zeolite-supported catalysts having higher Pt loadings is planned.

## Other Communications

J. Monroe, J. Dong, X. Gu, and J. Zhang, "Development of Novel Catalytic Membrane Reactors for Conversion of Biomass-Derived Carbohydrates to Hydrogen," accepted for the PacificChem 2005 Conference, Washington, DC.

# Reliability of Materials in MEMS: Residual Stress and Adhesion in a Micro Power Generation System

*80595*
*N. R. Moody*

## Abstract

There is currently no self-consistent method to quantify the interfacial fracture toughness of thin film systems that contain tensile stresses and compressive stresses. The project aims to make experimental measurements of adhesion that can be coupled with analytical solutions to determine interfacial fracture toughness. These results will provide computational model validation and design parameters, and lifetime analysis for microelectromechanical systems (MEMS) structures and thin films in microelectronics in the nation's stockpile. We will use three methods of interfacial adhesion: (1) compressed overlayer buckling, (2) four point bend testing, and (3) tensile "roll up" delamination using a stressed overlayer. The films tested will consist of both platinum films used in MEMS and gold films used in Sandia microelectronics systems. This project supports a fellowship for one PhD student, Marian (Molly) S. Kennedy, at Washington State University.

## Accomplishments

We deposited platinum films on a variety of microelectronic substrates both with and without compressed films. In this first year of support, we built a four point bend fixture at Washington State that is capable of performing the required tests. We demonstrated preliminary loading curves using a modified indentation system as the loading frame. This required development of sample fabrication and testing procedures, which are now similar to those used by other research groups. In particular, we addressed the loading frame stiffness so that no compliant adhesive is used in securing the samples. However, the loading frame used in acquiring the data is not capable of reaching low enough displacement rates. Therefore, we are using a new load frame with the fixture to reach the appropriate displacement rates (80 nm/s).

Finally, we indented compressively stressed overlayers of W on Pt. They generate buckles suitable for adhesion measurements. We are analyzing buckle indentations to quantify the adhesion between the platinum film and the oxide substrate with and without a Ti adhesion layer.

## Other Communications

M.S. Kennedy, N.R. Moody, and D.F. Bahr, "Investigation of Indentation Methods for Properties Determination in Hard Film – Soft Substrate Systems," presented and in *Proceedings of the Materials Research Society*, vol. 841, MRS 2004 Fall Meeting, Boston, MA, November 2005, pp. R12.7.1–6.

N.R. Moody, J.M. Jungk, M.S. Kennedy, S.V. Prasad, D.F. Bahr, and W.W. Gerberich, "Mechanical Properties of Wear Tested LIGA Nickel," presented and in *Proceedings of the Materials Research Society 2004 Fall Meeting*, Boston, MA, November 2004, vol. 841, pp. R7.8.1–6.

# Modeling River-Aquifer Interaction with Application to the Rio Grande

*80596*
*V. C. Tidwell*

## Abstract

This work will address 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. Work will focus on modeling the interaction between surface water and groundwater and its implications for resource management.

Regional groundwater models tend to be three-dimensional, 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.

Research for this work will include hydrologic modeling of the Rio Grande Project, extending from Caballo Dam in New Mexico to Fort Quitman, Texas, and includes diversion of surface water to Mexico at Juarez. Working with New Mexico State University, the Army Corps of Engineers, Bureau of Reclamation, and Paso del Norte Watershed Council, we will use these data for the formulation and calibration of a coupled hydrologic routing model and groundwater flow model. This model will initially examine flood routing and ultimately help to improve and manage operations of the Rio Grande Project.

## Accomplishments

We compiled flow data for the following sites along the Rio Grande between Caballo Dam, NM, and Ft. Quitman, TX, for the years 1975–2002:

- Major diversion sites
- Significant drain return flow sites
- Significant wasteway return flow sites
- Gauged river stations
- Wastewater treatment plant return sites

We found daily data for most of the sites; however, there were a few years for which some of the sites only had monthly data available.

We spent time learning the Riverware software in order to know both what type of data and hydrologic parameters are required for a model and then how to build that model.

We developed a conceptual model of the Rio Grande between Caballo Dam and the Rio Grande at El Paso. This conceptual model identified schematically the sites along the Rio Grande and their relative physical locations that would be included in the Riverware model.

The next step was to develop the hydrologic parameters required for the model. We found that the drain return flows are highly correlated to the diversions from the river, so we chose a regression model with autoregressive moving average (ARMA) errors as the technique to develop these relationships. To establish a methodology for developing these regression models, we selected the Rincon Valley portion of the Rio Grande as the test site to verify both the adequacy of the regression model technique and the ability to incorporate this technique into a Riverware model.

We developed a Riverware model for the Rincon Valley portion of the Rio Grande using monthly flow data, and regression models that define the relationships between the diversions at Arrey Canal and the return flow in the Garfield, Hatch, and Rincon drains.

# Kinetics and Mechanisms of Nanowire Synthesis

*80598*

*J. W. Hsu*

## Abstract

The goal of this project is to develop a fundamental understanding of the heteroepitaxial growth of semiconducting nanowires and nanowire arrays for potential applications in nanoscale electronic, photonic, and sensor devices. Our studies focused on understanding and controlling nanowire growth kinetics of silicon and germanium nanowire epitaxy by chemical vapor deposition (CVD), using the vapor-liquid-solid (VLS) growth technique, in our homebuilt ultrahigh vacuum (UHV) system. In these investigations, we demonstrated that the in situ, real time monitoring of nanowire nucleation and growth are extremely useful in understanding the nanowire growth kinetics. We also developed a new method to measure nanowire growth in real time using optical reflectometry. In this technique, previously used to monitor thicknesses during CVD uniform semiconductor layer growth, we treat the nanowire region as an effective dielectric layer. Based on measurements and modeling, we show that we have surprisingly good sensitivity to observe onset of nanowire growth and the evolving thickness of the nanowire growth region. As a result, we are able to quantitatively determine the nanowire nucleation times, the mean growth rate of nanowires, and the average lengths of the nanowires with good sensitivity. We expect this technique to be broadly applicable to the in situ control of nanowire heterostructure fabrication and to the understanding of the kinetics of semiconductor nanowire growth.

## Accomplishments

This research employed our home-built UHV CVD system to investigate Au seeded VLS growth of Si and Ge nanowires on Si(100) and Si(111). We developed an optical reflectometry technique for in situ monitoring during VLS nanowire growth, and demonstrated Si/Ge nanowire growth for linear and core-shell heterostructures. We can also achieve our Si and Ge nanowire synthesis at lower temperatures and pressures than usually employed in VLS nanowire CVD growth. Another result based on our in situ measurements is the discovery that a well defined nucleation time exists (from seconds to minutes) before nanowire growth begins. This phenomenon has not previously been observed, but can be understood since vapor-liquid-solid growth requires several steps before the onset of nanowire growth, most importantly before the supersaturation of the liquid seed eutectic. Our preliminary results show that the incubation time for nucleating nanowire growth increases as disilane pressure is reduced, clearly showing the role of precursor partial pressure and resulting chemical potential.

Key results during FY 2005:

(1) modification of a homebuilt UHV CVD system to enable in situ deposition of metallic seeds on UHV cleaned Si surfaces and demonstration of Au seeded epitaxial nanowire VLS growth from (111) silicon substrates

(2) development of an in situ optical scattering measurement capability and demonstration of the first optical reflectometry measurements during CVD growth of nanowires

(3) modeling of the optical reflectivity of growing Si nanowires based on an effective dielectric layer approach, enabling application of this new technique to determine nanowire nucleation times and growth kinetics

(4) preliminary demonstration of the growth of Si/Ge nanowire heterostructures using the in situ optical reflectometry technique

(5) first measurements of the nucleation times for Si nanowire growth and demonstration of the increase in nucleation times with decreasing CVD disilane precursor pressures

## Refereed

J.L. Taraci, M.J. Hÿtch, T. Clement, P. Peralta, M.R. McCartney, J. Drucker, and S.T. Picraux, “Method for Strain Mapping in Nanowires,” *Nanotechnology*, vol. 16, pp. 2365–2371, 2005.

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J.W. Dailey, J. Taraci, T. Clement, D.J. Smith, J. Drucker, and S.T. Picraux, "Vapor-Liquid-Solid Growth of Germanium Nanostructures on Silicon," *Journal of Applied Physics*, vol. 96, pp. 7556–7567, 2004.

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T. Clement, J.L. Taraci, J.W. Dailey, D.J. Smith, J. Drucker, and S.T. Picraux, "VLS Epitaxy of Si Nanowires: Kinetics and Morphology," presented at the American Vacuum Society 51st International Symposium, Anaheim, CA, November 2004.

J.L. Taraci, M.J. Hÿtch, T. Clement, J.W. Dailey, D.J. Smith, P. Peralta, M.R. McCartney, J. Drucker, and S.T. Picraux, "Strain Mapping in Nanowire Heterostructures," presented at the American Vacuum Society 51st International Symposium, Anaheim, CA, November 2004.

S.T. Picraux, R. Rosario, T. Clement, J.L. Taraci, J.W. Dailey, D. Gust, A.A. Garcia, and M. Hayes, "Light-Induced Contact Angle Switching on Nanowire Surfaces," presented at the American Vacuum Society 51st International Symposium, Anaheim, CA, November 2004.

T. Clement, S. Ingole, J. Drucker, and S.T. Picraux, "In Situ Studies of Semiconductor Nanowire Growth Using Optical Reflectometry," presented at the TMS Electronic Materials Conference, EMC2005, June 2005.

S.T. Picraux, T. Clement, J. Taraci, J.W. Dailey, D.J. Smith, J. Drucker R. Rosario, D. Gust, A.A. Garcia, and M. Hayes, "VLS Epitaxy of Si and Ge Nanowires: Kinetics, Morphologies, and Properties," presented at the Materials Research Society, Fall Meeting, November 29–December 2, 2005.

J. Taraci, R. Rosario, T. Clement, A.A. Garcia, D. Gust, M. Hayes, S.T. Picraux, B. Bunker, and J.E. Houston, "Nanoscale Switchable Surfaces," presented at the Center for Integrated Nanotechnologies (CINT) User Workshop, January 2005.

# Rapid Chemical Analysis Using Micropower Gas Chromatographic Columns and Latching Microvalves

*80599*
*A. Robinson*

## Abstract

Sandia has provided a fellowship to the University of Michigan to promote research in the areas of microfabrication science and engineering. The fellowship directly supports a graduate student during their doctoral studies.

This project seeks to develop a portable chemical analysis system capable of rapid analysis using low-mass gas chromatographic (GC) microcolumns and low power, latching microvalves. Rapid, low power chemical analysis is relevant in many fields, particularly for national security, where a fast response time from a battery powered, hand-portable system is critical. Examples of end users include first responders (Homeland Security) and war fighters (Department of Defense) concerned with rapid detection and identification of chemical weapons agents (CWAs) and toxic industrial compounds (TICs). This research has the potential to push existing portable GC performance by enhancing chromatographic resolving power while decreasing the overall analysis time. Accurately controlled thermal modulation of the GC columns will allow for improved peak separations that are not easily achieved using other separation and analysis methods. Applications include analysis of various analyte sets meeting requirements of multiple customers.

## Accomplishments

At the beginning of the fiscal year, we designed a low mass GC microcolumn, incorporating a buried circular microchannel. We sealed the channel using chemical vapor deposition (CVD). We then etched back the device to achieve minimal mass, which is advantageous for rapid thermal cycling during chromatography. During operation, the column heats quickly through integrated thin film resistors. We achieved separations of simple hydrocarbon mixtures using these columns. However, stress in the structural layer resulted in problems during release of the device and in its structural stability after release.

We subsequently investigated, simulated, and tested more robust processes and structures. We formed initial devices using stress-compensated oxide/nitride/oxide layers deposited by low pressure CVD (LPCVD). We successfully released and tested those; however, process repeatability and yield were major obstacles. On the other hand, oxynitride layers from LPCVD and plasma enhanced CVD, (PECVD) provided zero-stress structural layers, while improving both release of the devices and their stability. Next, we redesigned the column ports to significantly reduce dead volume issues.

We are also developing a low power microfabricated valve. The microvalve utilizes a thermo-pneumatic actuation mechanism coupled with electrostatic latching. The hybrid actuator provides high force and large throw while minimizing power, gas leakage, and sensitivity to particulate contamination. At the beginning of the fiscal year, the valve had a leak rate under $2E^{-2}$ sccm (standard cubic centimeters per minute), an open flow rate of 3 sccm at 100 Torr, a response time of 1 second using an energy input of 200 mJ, and a latching voltage of 40 V. However, latching was problematic.

We made progress on the microvalve during the year. We improved the model of the thermo-pneumatic actuator and used it to redesign the valve. The results predict tenfold improvements in speed and power consumption. In addition, we changed the structure of the valve slightly to improve its latching, lifetime, and ability to be integrated into the micro-GC system. At present, the modified valve and column designs are in fabrication stages. Once complete, we will fully characterize both devices and combine them in a pressure-tuned system to demonstrate rapid, low power GC separations.

## Refereed

M. Agah, J.A. Potkay, G.R. Lambertus, R.D. Sacks, and K.D. Wise, “High-Performance Temperature-Programmed Microfabricated Gas Chromatography Columns,” to be published in *IEEE Journal of Microelectromechanical Systems.*

C.-J. Lu, W.H. Steinecker, W.-C. Tian, M. Agah, J.A. Potkay, M.C. Oborny, J. Nichols, H. Chan, J. Driscoll,

R.D. Sacks, S.W. Pang, K.D. Wise, and E.T. Zellers, "First Generation Hybrid MEMS Gas Chromatograph," to be published in *Lab On A Chip*.

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## Other Communications

J.A. Potkay and K.D. Wise, "An Electrostatically Latching Thermopneumatic Microvalve with Closed-Loop Position Sensing," presented and in *Proceedings of the 18th IEEE Conference on Micro Electro Mechanical Systems*, Miami, FL, January 2005, vol. 1, pp. 415–418.

# A Generalized Simulation Environment for Particulate Systems

*80600*
*B. K. Cook, S. M. Johnson*

## Abstract

The physics of granular materials are important in numerous industrial problems. For example, the majority of drugs that are in use today are produced as dry powders. Our limited understanding of particulate systems, however, prevents the pharmaceutical industry from designing effective processing techniques to regulate the mixing and formation of powder based tablets. Solutions to other engineering challenges, such as bulk materials transport in mining and sand production from oil wells, are also hampered by our inability to accurately predict the mechanical behavior of granular systems.

This project addresses this through the development of an extensible three-dimensional (3-D) modeling framework for particulate systems based on the discrete element method (DEM). DEM is a suite of numerical algorithms that allows for the efficient modeling of granular materials as a collection of idealized rigid bodies that interact through surface contacts and bonds. Numerically akin to molecular dynamics, the method can also be extended to include long range interactions between particulate grains such as those that arise in powders from electrostatic forces. For computational expediency, most current DEM models use spherical elements to represent particles, which is a poor approximation of the shape of many natural grains. We will address this deficiency through the implementation of computationally compact representations for non-spherical shapes that require minimal effort for contact detection and resolution. We will develop an extensible software framework to support interactive model definition and real time visualization.

We will perform this project in collaboration with researchers from the Massachusetts Institute of Technology (MIT). In addition to the aforementioned particulate applications, DEM is also useful for understanding and modeling the mechanics that govern the behavior of composite materials (e.g., rock and concrete) undergoing large deformations, which is important in problems such as penetration, shock-structure interaction, and blast damage.

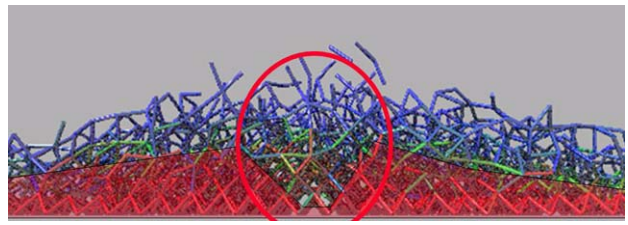

*Illustration of visualization technique for showing contact age through the cross section of a granular heap composed of dry, poly-disperse spherical grains. The coloring is normalized to show oldest contacts in red and youngest contacts in blue.*

## Accomplishments

Research in FY 2005 focused on the implementation, validation, and verification of a particulate system simulator, along with supporting development of DEM algorithms. We also performed several numerical experiments with the DEM simulator focusing on two key areas: simulation of pharmaceutical powder blending and analysis of stress fabric development in granular heaps.

Algorithmic development furthered work on three issues:

(1) We augmented ellipsoidal representations to include a new asymmetric ellipsoid contact resolution algorithm based on the fundamental concepts employed in earlier work on axially symmetric ellipsoids.

(2) We developed a polyhedral boundary contact resolution algorithm that reduces the dimensionality of the test by one over previously used methods.

(3) We formulated a representation of multi-scale, multi-physics surface behaviors for capturing both emergent properties such as cohesion and physical properties such as surface asperities. The formulation is based on a multi-resolution discretization of the convex surface of an idealized grain geometry. The flexibility of the approach allows for the layering of several different types of data onto the surface, along with fast lookup performance for resolving the surface data into physical behavior.

We integrated all major DEM algorithms, including numerical quadrature, contact resolution, and neighbor-sorting, into a testing and benchmarking

framework. Also, we extended, validated, and compared the proposed rotational integration algorithm against other algorithms through the testing and benchmarking framework.

With collaborators in the Department of Chemical Engineering at MIT, we are verifying the DEM3-D framework against experimental results obtained both at the microscopic and mesoscopic levels. The approach uses parameters obtained from atomic force microscopy experiments of powder particle separation tests to calibrate the Johnson, Kendall, Robert's theory (JKR) model of cohesion. We then use this model, with the calibrated parameters, as a contact law in a DEM simulation. We modified the model from a purely JKR implementation by adding a log normal stochasticity on a per contact basis. We then compare the homogeneity of the mixture at the apex cap of the blender with the same measurement using light induced fluorescence on a bench scale blender. This collaboration resulted in good qualitative agreement between simulation and experiment.

A significant effort over the past year focused on data analysis and data archiving for DEM simulations. Typically, the focus of DEM research is to develop more efficient algorithms to allow the computation of larger simulations; however, the associated need of archiving and interpretation of results is often overlooked. We proposed and prototyped a strategy for archiving and then analyzing the data produced by a DEM simulation in the DEM 3-D framework. The work integrated a commercially available database to the framework. We developed a stand-alone application to merge the data visualization capabilities of the simulator with a custom data analysis framework structured around aggregated queries.

Using this framework, we demonstrated a unique capability to investigate the history of stress fabrics in a granular system. By using the lifetime of contacts, we were able to determine areas of fabric damage during the construction of granular heaps that, in our small sample of systems, lead to a cratering effect in the center of the pile. In turn, this leads to the construction of stable structures of grains along the crater edge. As the material fills in the crater, the old stable formations at the crater edge form the base of an arch that helps to distribute stresses away from the center of the heap. The effect of this phenomenon on the base stress emerges as a dip in the stress at the center of the heap. The hypothesis also seems to hold under the known conditions that affect the formation of a stress dip, including the degree of poly-dispersity of the grains and asphericity.

## Refereed

S.M. Johnson, J.R. Williams, and B.K. Cook, "Contact Detection Algorithm for an Ellipsoid Approximation for Discrete Element Modeling," *Engineering Computations*, vol. 21 (N2-4), pp. 215–234, 2004.

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S.M. Johnson, J.R. Williams, and B.K. Cook, "Contact Detection Between Axially Asymmetric Ellipsoids in Discrete Element Modeling," presented and in *Proceedings of the Computational Fluid and Solid Mechanics 2005*, at the Third MIT Conference on Computational Fluid and Solid Mechanics, Cambridge, MA, June 2005, vol. 1, pp. 268–270.

# Automated Assembly of Microscale Devices

*80601*
*J. F. Jones*

## Abstract

Microelectromechanical systems (MEMS), with their ability to integrate moving mechanical parts, electronics, and other functions inexpensively, have 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 related technologies are drastically limited in several important ways. The key problem is the inability to form true three-dimensional (3-D) structures, of the needed complexity, composed 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 products based on microscale parts will be produced. This research works towards addressing the main robotic issues regarding the assembly of complex 3-D 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 apply 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 intends to provide the basis for modular and scalable micro factories that will enable the economically viable assembly of complex 3-D microscale mechanisms. It will also enable integration of hybrid microsystems relevant to nuclear weapons applications.

## Accomplishments

We identified the existing Carnegie Mellon University minifactory platform as the platform to conduct experiments and evaluations in order to determine the issues and technological gaps associated with using this architecture for assembly of small lots of 3-D microscale mechanisms. The goal for FY 2005 was to identify and develop key minifactory components to enable these experiments. The component identified for development was a new end effector agent that possessed the high resolution optics necessary for microscale visual servoing, and a tweezing mechanism capable of manipulating parts in the range of tens of microns to a few millimeters.

The optic system consists of a Mitutoyo long range objective and a charge-coupled device camera. The long range objective permits the optical system to focus on a point several centimeters from the objective lens. This capability allows space for the gripper to be placed between the lens and the working surface. The concept of operation is that a parts carrier positions a part or subassembly to a defined location within the view of the optic system. The optic system then commands the parts carrier to adjust its location to correct for variability in the process.

The gripping mechanism consists of commercially available precision tweezers with an integrated actuator that permits automatic control of the tweezers. The device improves upon a similar microgripping mechanism developed at Sandia by employing a voice coil actuator. We designed and fabricated this new end effector agent. Future work will focus on performing experiments to assess its ability to assemble 3-D microscale mechanisms.

# MEMS Reconfigurable Intelligent RF Circuits

*80602*

*C. W. Dyck, G. M. Kraus, C. D. Nordquist*

## Abstract

Microwave power amplifiers are principle components in communication and radar systems. The addition of radio frequency (RF) microelectromechanical systems (MEMS) switches enables reconfigurability and allows for multi-functional performance with significant size and cost reduction. One example is a power amplifier that switches between a linear class-A mode of operation (useful in communication) and an ultrahigh efficiency class-E mode (useful in high power radar). Currently, due to very different requirements, amplifier circuits in communication and radar systems are separate. Arrays of these switched amplifiers would allow these two systems to be combined. We recently demonstrated this amplifier and achieved 49% power added efficiency (PAE) in the class-E mode and a gain and output power of 11 dB and 20 dBm in the class-A mode. The slight loss of the MEMS switches was responsible for a 3% to 4% reduction in PAE from a standard untuned class-E amplifier on the same substrate.

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 no method exists to determine the optimal interstage impedance in these multistage amplifiers. A MEMS 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 they could be removed and used solely as a measurement tool. We investigated this approach on paper during the previous year and expect it to be the focus of the upcoming year in collaboration with the University of Colorado, Boulder.

## Accomplishments

In FY 2005, we investigated and characterized basic switched impedance networks. We fabricated these networks using RF MEMS switches placed in a microstrip shunt stub to change the physical length of the stub, thereby changing the impedance of the network. We also varied the switch position along the length of the stub to study the effect of position on the overall insertion loss of the network. We compared these switched networks to measured microstrip networks in order to understand the cost-benefits of switched networks to conventional designs. In parallel, we constructed and characterized both class-A and class-E amplifiers for direct cost-benefit comparison with the switched-mode amplifier. We also used these amplifiers to determine a maximum tolerable decrease in efficiency (due to the additional insertion loss a switched network has compared to its microstrip equivalent) for the benefit of reconfigurability. The switched network study and amplifier specifications allowed the switched-mode amplifier, with two switched shunt stubs, to be designed with a calculated decrease in efficiency of less than 5% compared to the measured independent class-A and class-E amplifiers.

Following this characterization, we integrated the amplifier with the MEMS tuning network to demonstrate the class-A to class-E switched mode amplifier. We attached the amplifier to the MEMS network with a low outgassing, space-qualified epoxy to prevent contamination of the switch contacts. We achieved excellent experimental results during recent characterization. The class-A mode of operation achieved a PAE of 24% at 1 dB compression, a drain efficiency of 27%, gain of 11 dB, and maximum output power of 20 dBm. The class-E non-linear mode of operation achieved PAE of 49%, drain efficiency of 58%, and maximum output power of 21 dBm. The two MEMS switches used to reconfigure the amplifier between class-A and class-E modes contributed less than 0.3 dB of additional loss to the amplifier. We extracted this value from comparisons between measurements of networks

both with and without MEMS. We are currently measuring the intermodulation distortion product of the reconfigurable amplifier.

## Refereed

P.J. Bell, Z. Popovic, and C.W. Dyck, "MEMS-Switched Class-A-to-E Reconfigurable Power Amplifier," to be published in *Proceedings of the 2006 MTT Radio and Wireless Symposium*.

# Bayesian Inference for Inverse Problems, Model Structure, and Uncertainties

*80603*

*Y. Marzouk, H. N. Najm, L. A. Rahn*

## Abstract

Inverse problems are of great relevance to science and engineering. While progress in detailed first principles modeling of physical systems has been enormous, it has exposed the challenging and complementary tasks of 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 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 of great importance to homeland security response to current risks of bio-agent 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 structures may fundamentally enhance knowledge of crucial aspects of cellular function by elucidating pathways between normal and diseased cellular states, thereby clarifying responses to pathogens and toxins. Therefore, the work will also impact Sandia's portfolio in biotechnology and molecular biology. Principal Investigator Youssef Marzouk is a recipient of the post-doctoral Truman Fellowship Award.

## Accomplishments

Work in FY 2005 first saw the formulation of a Bayesian approach for continuous inverse problems, specifically contaminant source inversion under convection and diffusion. The present Bayesian formulation yields a joint posterior probability density for the parameters describing a finite set of sources. We demonstrated a Bayesian solution to the source inversion problem for a single stationary source with a time dependent strength under transient diffusion. In the process, we demonstrated the ability of the Bayesian formulation to quantitatively incorporate sensor noise, prior information, and solution non-uniqueness. We also extended this Bayesian approach to include explicit treatments of parametric and non-parametric uncertainty in the forward model, thus accurately quantifying confidence in inverse solutions from imperfect, uncertain, or reduced-order physical models.

The core computational challenge associated with a Bayesian formulation remained the cost of evaluating high-dimensional integrals over the posterior density, particularly when each sample or quadrature point requires solution of the forward problem. To address this difficulty, we formulated a stochastic version of the forward problem, using spectral expansions (specifically, polynomial chaos representations) to propagate the entire range of prior uncertainty through the time dependent scalar transport equations. This construction thus replaces the enormous cost of repeated forward problem evaluations with an efficient spectral representation of the model outputs.

We first demonstrated this polynomial chaos (PC) reformulation of Bayesian inference on simple algebraic inverse problems, then on a time dependent, diffusive source inversion problem. In both of these contexts, we coupled the PC approach with Monte Carlo and state-of-the-art Markov chain Monte Carlo sampling strategies in order to evaluate posterior expectations and to compute marginal distributions. For complex inverse problems such as source inver-

sion, our new technique offers dramatic gains in computational efficiency: approximately 2–3 orders of magnitude with no appreciable loss of accuracy. The present work thus significantly extends the range of feasibility of Bayesian inference for large scale inverse problems.

## Refereed

Y.M. Marzouk, H.N. Najm, and L.A. Rahn, "Stochastic Spectral Methods for Efficient Bayesian Solution of Inverse Problems," presented and in *Proceedings of the Bayesian Inference and Maximum Entropy Methods in Science and Engineering*, San Jose, CA, August 2005.

Y.M. Marzouk and A.F. Ghoniem, "Vorticity Structure and Evolution in a Transverse Jet," submitted to *UK Journal of Fluid Mechanics*.

Y.M. Marzouk, H.N. Najm, and L.A. Rahn, "Stochastic Spectral Methods for Bayesian Inference in Inverse Problems," to be presented at the CSDA 2005 (3rd IASC World Conference on Computational Statistics and Data Analysis), Amsterdam, Netherlands, October 2005.

Y.M. Marzouk and A.F. Ghoniem, "K-Means Clustering for Optimal Partitioning of Parallel Hierarchical N-Body Simulations," presented at the 16th International Conference on Domain Decomposition Methods, New York, NY, January 2005.

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D. Wee, Y.M. Marzouk, and A.F. Ghoniem, "Lagrangian Simulation of a Jet in Crossflow at Finite Reynolds Number," presented and in *Proceedings of the AIAA Aerospace Sciences Meeting 2005*, Reno, NV, January 2005, CD-ROM.

D. Wee, Y.M. Marzouk, and A.F. Ghoniem, "K-Means Clustering and Diffusive Interpolation for High-Resolution Vortex Particle Methods," presented at the APS Division of Fluid Dynamics, 57th Annual Meeting, Seattle, WA, November 2004.

R.L. Speth, Y.M. Marzouk, and A.F. Ghoniem, "Impact of Hydrogen Addition on Flame Response to Stretch and Curvature," presented and in *Proceedings of the AIAA Aerospace Sciences Meeting 2005*, Reno, NV, January 2005, CD-ROM.

Y.M. Marzouk and A.F. Ghoniem, "Vorticity Transformation Mechanisms and Mixing in a Transverse Jet," presented at the APS Division of Fluid Dynamics, 57th Annual Meeting, Seattle, WA, November 2004.

R.L. Speth, Y.M. Marzouk, and A.F. Ghoniem, "A Quasi-One-Dimensional Unsteady Laminar Flame Formulation with Independent Strain Rate and Curvature," presented and in *Proceedings of the Third MIT Conference on Computational Fluid & Solid Mechanics*, Cambridge, MA, June 2005, vol. 3.

## Other Communications

Y.M. Marzouk, "Towards Efficient Bayesian Solution of Inverse Problems," presented at the Workshop on Large-Scale Robust Optimization, Santa Fe, NM, September 2005.

Y.M. Marzouk and H.N. Najm, "Polynomial Chaos Acceleration of Bayesian Methods for Solving Inverse Problems," to be presented at the SIAM Conference on Parallel Processing for Scientific Computing, Philadelphia, PA, February 2006.

# Atomistic Modeling of Nanowires, Small Scale Fatigue Damage in Cast Magnesium, and Materials for MEMS

*80604*
*J. A. Zimmerman*

## Abstract

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 compose 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, Galvanoformung, 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 objective of the proposed work is to understand material characteristics in relation to mechanical properties at the nanoscale and microscale in these promising new material systems. We will conduct research at the University of Colorado at Boulder to employ tightly coupled experimentation and simulation to study damage at various material size scales under monotonic and cyclic loading conditions. We will accomplish experimental characterization of nano/micro damage by novel techniques, such as in situ environmental scanning electron microscopy (ESEM), 1 MeV transmission electron microscopy (TEM), and atomic force microscopy (AFM). New simulations to support experimental efforts include modified embedded atom method (MEAM) atomistic simulations and single crystal micromechanical finite element simulations.

## Accomplishments

The past year focused on three topics. Critical findings in these three areas are summarized below.

- Topic 1: Fatigue of Cast Mg

We performed the final leg of this section of the work in year four. The final work focused on the quantification of fatigue crack growth rates of microstructurally small cracks. These data constitute the first quantitative small fatigue crack growth study in environment using ESEM, and provide quantitative input for multi-length scale modeling efforts in cast Mg.

- Topic 2: Atomistic Simulations

Work in this arena focused on the plastic properties of the NiTi alloy nanowires in collaboration with Sandia, CA. We examined the effects of wire size, orientation, and structure on producing nanowires in a systematic manner. In particular, we demonstrated how various experimentally observed wires, such as single atom chains, multi-shell helical wires, and <110> wires, deform. The results constitute a fundamental understanding of the yield behavior of nanowires as a function of size scale.

- Topic 3: Properties of Thin Films for Microelectromechanical Systems (MEMS)

Work in this area focused on thin Au films on Si microcantilevers. We performed thermomechanical studies, nanoindentation, spectroscopy, and microscopy. The results demonstrate how the films behave and evolve in their as-deposited state at various temperatures.

With the final year of funding, two students will be supported to finish their PhD thesis topics; both research areas are related to the small scale deformation of materials. Topic 1 is the deformation of metallic nanowires, and this is a spin off of the funded work on the properties of thin films for MEMS. The objective of this project is to use MEMS structures and nanoindentation to examine the deformation of chemically grown nanowires. Topic 2 is the nanoindentation of NiTi shape memory alloys. The objective of this project is to understand the fundamentals of stress induced phase changes in shape memory alloys at nanometer length scales.

## Refereed

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D. Miller, C.F. Hermann, H.J. Maier, S.M. George, C. Stoldt, and K. Gall, "Intrinsic Stress Development and Microstructure Evolution of Au/Cr/Si Multilayer Thin Films Subject to Annealing," *Scripta Materialia,* vol. 52, pp. 873–879, 2005.

D. Miller, C.F. Hermann, H.J. Maier, S.M. George, C. Stoldt, and K. Gall, "Thermo-Mechanical Stability of Thin Film Multilayers: Part I Mechanical Behavior of Au/Cr/Si Microcantilevers," submitted to *Thin Solid Films*.

J. Diao, K. Gall, M.L. Dunn, and J.A. Zimmerman, "Atomistic Simulation of the Yield of Au Nanowires," submitted to *Acta Materialia*.

D. Miller, C.F. Hermann, H.J. Maier, S.M. George, C. Stoldt, and K. Gall, "Thermo-Mechanical Stability of Thin Film Multilayers: Part II Microstructure Evolution," submitted to *Thin Solid Films*.

# Design, Analysis, and Control of MEMS Devices for Micromanipulation Tasks

*80667*
*J. J. Allen*

## Abstract

Control systems are regularly used in the macro world to ensure proper system operation despite disturbances from noise or uncertainties. The objective of this research is to develop and apply passive and active control techniques to microelectromechanical system (MEMS) devices for high precision performance. The specific application is electrostatic micromirrors that currently suffer from electrostatic pull-in instability and inconsistent performance due to fabrication variations. We will use passive control, achieved through the design and development of nonlinear mechanical components, to eliminate undesirable system behavior such as electrostatic pull-in. We will accomplish active, closed-loop control to ensure very precise and accurate operation, despite the effects of design and fabrication uncertainties, by incorporating on-chip piezoresistive sensing.

Electrostatic actuators suffer from an instability known as "pull-in" that occurs when the electrostatic force exceeds the mechanical restoring force of the device. This instability, while advantageous for digital designs in which only two stable positions are required, is a limitation for devices that require continuous motion over the full actuation range. Another phenomenon associated with this instability is that once the mirror has pulled-in, it will not return from this position until the actuating voltage has been reduced below a threshold voltage, resulting in hysteresis. The behavior of electrostatic instability and hysteresis can be predicted through system modeling, and new designs may be developed to exploit mechanical nonlinearities for improved performance. In addition to controlling the mirror across the instability point, feedback control design for high precision, micromanipulation can be realized through the development of on-chip piezoresistive sensing. The successful completion of this work will enable more precise control of MEMS devices than is presently available. This will provide capability for micro-optics and micro-robotic applications that is not currently available.

## Accomplishments

During FY 2005 we accomplished the following tasks:

- We experimentally characterized and evaluated the rotational grating element with integrated piezoelectric sensors. We proved the design techniques for fabricating the piezoresistors adequate for this application.
- We experimentally characterized and evaluated the rotational grating element using interferometry. The data obtained were rotation angle versus voltage applied. The device obtained 19 degrees of rotation at maximum voltage.
- We obtained and correlated an analytical model of the rotational grating element with the experimental data obtained.
- We incorporated non-linear mechanical linkages that alter the restoring force of the electrostatic micro mirror such that the pull-in instability is delayed or eliminated. We measured the rotational grating elements with the nonlinear stiffness via interferometry.
- We characterized the piezoresistive sensing network and obtained the gauge factor. We will incorporate this information into the next system design.
- We developed an analytical model for a vertical-comb-drive actuated micro mirror. We developed the model using finite element analysis and analytical techniques to determine the equilibrium positions of the mirror, pull-in, and hysteresis.

## Refereed

G.A. Roman, J.R. Bronson, G.J. Wiens, J.F. Jones, J.J. Allen, "Design of a Piezoresistive Surface Micromachined Three-Axis Force Transducer for Microassembly," presented and in *Proceedings of the IMECE 2005, Proceedings*, Orlando, FL, November 2005, p. 81672.

J.R. Bronson, G.J. Wiens, J.J. Allen, "Modeling and Alleviating Instability in a MEMS Vertical Comb Drive Using a Progressive Linkage," presented and in *Proceedings of ASME International Design Engineering Technical Conference*, Long Beach, CA, September 2005, p. 84217.

# Developing Novel Scaffolds for Biological Molecules by Solving the I-QSAR Problem Using the Signature Molecular Descriptor

*82854*

*J. M. Faulon, D. P. Visco Jr.*

## Abstract

The broad field of quantitative structure activity relationships (QSARs) allows researchers in many areas to use such dependencies to correlate properties with particular features of the compounds examined. After this information is gleaned, the next step is to use the results on the relative importance of some features to predict information about compounds that have not been examined in this way. The hope is, of course, that researchers can develop better compounds that have optimal desired properties. Unfortunately, the limiting step in the procedure is how to best quantify the particular features of a compound's structure. Work by Professor Visco, in collaboration with Sandia, made great strides in addressing this problem through the introduction of a molecular descriptor, called Signature, which quantifies a molecule's two-dimensional (2-D) structure. Previous work focused on using Signature to assess the biodiversity of a small database (~100 HIV-1 protease inhibitors) and a large database (~30,000 AIDS antiviral drugs). Work also focused on the study of the degeneracy/information content of Signature relative to dozens of other popular descriptors. As this technique matures, certain algorithmic bottlenecks become apparent as we fine tune this methodology against a variety of different problems. Since the longer term goal of this work is to develop an approach that goes from "data set" to "focused library," it is important to flesh out all of these areas in the earlier stages of the research. To this end, current work will focus on these issues via several concurrent studies. Ultimately, once focused libraries are generated, experimentation will occur (as available) to provide a feedback mechanism to the algorithm.

## Accomplishments

In this initial year of this work, we performed several concurrent studies in order to expose bottleneck issues associated with the algorithm. The studies performed were on four systems: Cyclooxygenase-2, Dihydrofolate Reductase, Candida Albicans, and Gamma-Secretase. Lessons learned: (1) Some systems cannot be described by a multiple-linear regression QSAR and different (i.e., non-linear) models are required. (2) To limit the combinatorial explosion of a problem, partition the problem based on reasonable boundaries (i.e., based on strains used or cell lines). (3) The two major bottlenecks are the generation of solutions from the Diophantine equations and the generation of linear combinations of solutions from the basis vectors. During the year, we developed an alternate algorithm as a work-around for these bottlenecks. Current collaborative work at both Sandia and Tennessee Tech are aiding these accomplishments.

This work also resulted in the following:

- the graduation of two students of distinction in the major undergraduate thesis projects
- ongoing work for two Master's thesis students
- three invited presentations at different universities across the United States
- the "New Faculty Research Award" given by the Southeastern Section of the American Society of Engineering Education (ASEE).
- two student awards at the regional American Institute of Chemical Engineers meeting and the sectional ASEE meeting

## Refereed

D.C. Weis, J.L. Faulon, and D.P. Visco Jr., "The Signature Molecular Descriptor. 5. The Design of Hydrofluoroether Blowing Agents Inverse-QSAR," to be published in *I&EC Research.*

# Dynamics of Particulate Film Formation on Surfaces During Coating

*84317*
*P. R. Schunk*

## Abstract

We computationally studied the stability of various two-dimensional (2-D) arrays of bidisperse mixtures of stabilized nanoparticles through a melting simulation by employing the Metropolis algorithm for determining surface diffusion. In our previous work, we studied the stability of bidispersed monolayers of thiol-stabilized gold nanoparticles with a size ratio of 0.375. We found that interparticle forces were essential to stabilize the LS (the two-dimensional NaCl analog) lattice at the experimentally determined surface coverage. We extended our study to determine the conditions necessary to form stable LS2, LS4, and LS6 lattices, which have yet to be observed. Using a simple design rule that involves matching the distances between either large-large particles and large-small particles or large-small particles and small-small particles to correspond to the respective potential minima leads to predictions for size ratios that will form each desired lattice. Choosing constant values for the other parameters that govern the problem, we predict stable LS2, LS4, and LS6 lattices at relatively low surface coverages. Additional simulations show that the LS, LS2, and LS6 lattices are indeed stable structures at their predicted surface coverage, whereas the LS4 lattice is a metastable structure at the predicted surface coverage. This study may be used as a guide for experimentalists in their search for these novel structures. Additionally, we created a molecular dynamics simulation in a step toward understanding the effect of hydrodynamics on the microstructural development of nanometer to micron sized particles into 2-D arrays.

## Accomplishments

The production of extremely small structures, beneath the limits of modern-day lithographic techniques, is an area of intense interest these days. Self-assembly or “bottom up” methods of manufacture are being explored as an alternative means for forming nanoscopic structures as opposed to “top down” direct patterning methodologies. Ordered patterns of nanoparticles could be used directly as high-density magnetic media if the particles were ferromagnetic, or indirectly as templating agents. Other potential applications for these ordered arrays include photonic devices and meta-materials.

Experiments found in the literature showed that thiol-stabilized gold particles, with distinct bimodal size distributions, spontaneously order into 2-D superlattices. It was thought that hard-sphere interactions control the order-disorder transition, but we found that hard-sphere entropic ordering does not occur in these systems. We showed that the LS lattice was stable at the experimentally observed surface coverage if interparticle interactions in the form of van der Waals, osmotic, and steric forces were included.

We extended our simulations in search of stable 2-D LS2, LS4, and LS6 lattices. We are able to estimate the size ratio and surface coverages that will lead to each desired stable lattice by matching two of the three distances, corresponding to the pair-wise potential minimum (i.e., small-small particles, large-small particles, and large-large particles), to the geometry of each lattice structure. Melting simulations were then run for each lattice based upon the predicted size ratio and surface coverage, and in the cases of the LS, LS2, and LS6 lattices, resulted in stable structures. In the LS4 case, the slight adjustments for the parameters had to be made to achieve a stable structure.

Additionally, we performed simulations in which we filled half of the periodic cell with the designated lattice and the other half with random packing. In all, we maintained the total surface coverage. Based upon the movement of the interface, we determined that the LS, LS2, and LS6 lattices are stable states, while the LS4 lattice is a metastable state. The design rules, as validated by simulations, should be useful to experimentalists in searching for these new structures.

In addition to the Monte Carlo simulations performed, we created a molecular dynamics program that will follow the self-assembly of nanometer to micron sized particles into 2-D arrays. Relevant forces included in this preliminary simulation are simple repulsive forces between particles, drag forces, and a linear shearing force. This simulation is a step toward understanding how hydrodynamic forces offer an interesting new way to sample configuration in place of the limited thermal forces route that require the interparti-

cle energies to reside around only a few kT. By studying these effects, we will gain a more complete view of the self-organizing process.

## Refereed

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B.D. Rabideau and R.T. Bonnecaze, "Computational Predictions of Stable 2-D Arrays of Bidisperse Particles," to be published in *Langmuir.*

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R.T. Bonnecaze, "Self-Assembly of 2-D Arrays of Bidisperse Particles," presented at the ISCST Symposium, Rochester, NY, 2004.

B.D. Rabideau, "Simulation of the Self-Assembly of 2-D Arrays of Bidisperse Particles," presented at the AIChE Annual Meeting 2004, Austin, TX, November 2004.

# File System Performance Optimization for Supercomputing Applications

*85512*
*R. A. Oldfield*

## Abstract

Scalability demands on the file systems of future supercomputers are giving rise to new system level technologies. The purpose of this work, in conjunction with the Georgia Institute of Technology, is to explore and experiment with ways in which applications interface to make use of future scalable file systems and to address 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' central processing unit (CPU) 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 purposes are: (1) to enrich data sources with application specific functionality that manages the data streamed on behalf of individual clients, and (2) to take advantage of nodes "in between" the source and the sinks to perform processing and data movement actions that benefit sinks and reduce resource needs. Essentially, by moving transformation operations "into" logical network overlays, by combining identical operations and reducing composite CPU load, and by placing operations according to the CPU resources and network bandwidth available, we can attain overall improvements in the quality of service experienced by applications.

## Accomplishments

Large volume data streaming can be made more efficient by applying "in transit" operations on the streamed data. This project is developing automatic methods for (1) generating the code applied to data streams from higher level declarative specifications, (2) deploying such code onto overlay networks mapped across machines, and (3) optimizing and re-optimizing code and deployment, jointly, to better meet current end user needs and platform resources. Our accomplishments for FY 2005 include the following:

(1) We identified basic issues related to decomposition of data transformations into discrete operations. In particular, there are trade-offs associated with the expressiveness attained by separating dependent operations compared to the performance gained by combining such operations into indivisible elements. For example,

(a) The granularity and richness of operations may affect optimization opportunities. While richer operations require fewer transforms, more granular operations provide more explicit opportunities for optimization. The cost is complexity of the operations and potential ordering. This exposes the following broader questions: What kinds of operations should be supported natively in a transformation language, and how do we best describe operations that are beyond the standard set? Should there be a mix of declarative and operative code? How do we handle imperative constructs?

(b) It is unclear whether it is better to combine identical operations or duplicate them. With multiple requests for the same file, it is tempting to always combine common transformation operations. Depending on access patterns of the clients, keeping separate copies of identical transforms may allow better data flow and more efficient end-to-end performance for all clients.

(c) As clients are added and removed for a file stream, at what performance degradation level is it worth re-deploying the overlay network? How can that be calculated? We are now studying these trade-offs experimentally with sample molecular dynamics streaming data and analysis and display operations applied to such data. Future extensions will examine richer relationships and deployment issues.

(2) We designed and evaluated techniques for creating dynamic type and transformation information for nodes based on the incoming data type, outgoing data type, downstream transformation requirements, and transformation operations present on the node. With typed data streams based on file metadata, it is desirable to maintain the strict type information as the data

flows from the server to the client. Essential to this is the need to generate intermediate types. They are based on the incoming type, propagated elements necessary for downstream nodes, and transforms applied at the current node. These intermediate types temporarily include a propagation structure for source elements required for downstream transformation. We developed this type generation technique for all intermediate stages in the data stream.

(3) We explored techniques for the proper distribution of the transformation operations in the presence of various restrictions and conditions on the system. With varying performance requirements for file access, it is essential that any optimization system be flexible and adapt to the client's needs. To this end, we developed two deployment schemes that change the organization of the transformations based on data size and end-to-end performance.

We completed an experimental evaluation and research report on this research.

# Analysis of Bead Attached Ion Channels on Optically Addressable Microfluidic Electrode Arrays

*86358*

*S. M. Brozik, G. D. Bachand*


## Abstract

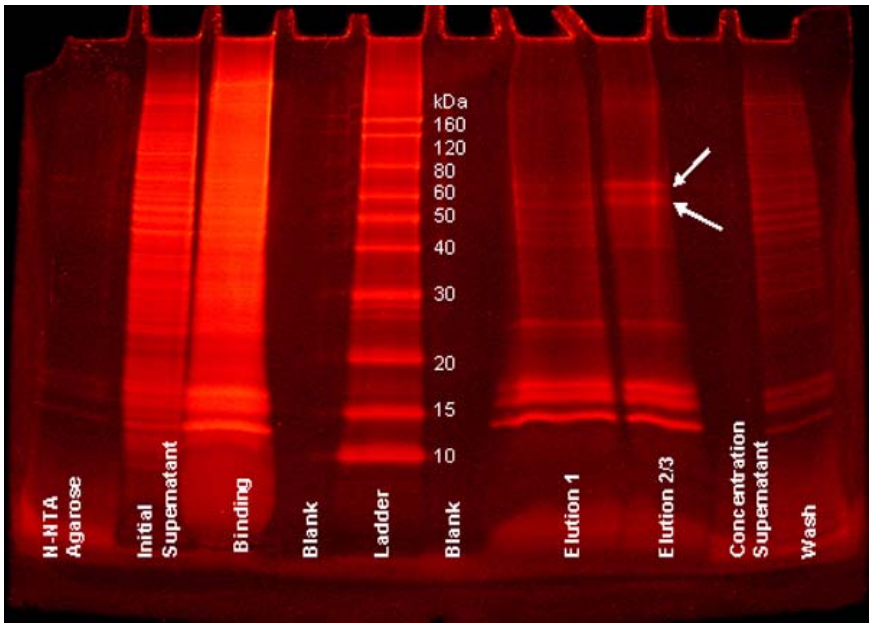
In order to better understand the fundamental structural dynamics of membrane-bound ion channels and membrane bound proteins in general, we are developing new solid-supported lipid bilayer platforms in which single membrane bound proteins can be electrochemically and optically interrogated. We demonstrated the capabilities of two of these types of systems by extracting the full thermodynamic properties of gramicidin ion channel formation in supported lipid bilayers, and recently demonstrated that the internal volume of bilayer-coated nanoporous silica beads remains excluded from bulk solution until the introduction of a poration medium. Combining these two observations provides the opportunity to use bilayer-coated porous silica beads as an ideal biological platform for the study of membrane-bound structures.

We are currently expanding this work to include more complicated membrane bound structures such as the light activated proton pump, bacteriorhodopsin, the ligand-gated ion channel, and the serotoinin type 3 receptor. We will achieve this via detergent reconstitution of these structures into small unilamellar vesicles (SUVs) followed by dialysis and exposure to porous silica beads. Confocal fluorescence imaging of fluorescently labeled protein confirms the presence of the protein, and a fluorescence recovery after photobleaching (FRAP) experiments determines the diffusion constant in this matrix. We will achieve confirmation of protein function and membrane orientation by loading porous beads with pH or calcium ion sensitive dyes prior to bilayer formation and monitoring the fluorescence after exposure to light or appropriate ligands. 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. They also show promise for new chip-based sensor technology.


## Accomplishments

(1) Design and construction of a microfluidic chip-based platform for simultaneous optical and electrochemical interrogation of membrane bound structures:

We modified the Si:SiN chips with 12 micron thick poly(vinyl formamide) thin films. We subjected the thin films to 200 µJ pulses of ultraviolet (UV) laser




*Expression and purification of the serotonin receptor.*

irradiation from a nitrogen laser focused through a 40x microscope objective to produce circular apertures with diameters ranging from 2–15 microns. Positioning of bilayer-coated nanoporous silica beads over these apertures produced gigaohm seals in ~10% of attempts. In order to increase this success rate, we are modifying the current design to include suction to hold the beads over the aperture.

(2) Expression and purification of the ligand gated ion channel, the serotonin receptor (5HT3R):

We expressed histadine tagged 5HT3R in a mammalian cell line and purified it. We used SDS-PAGE to confirm the presence of the receptor that has a molecular weight of ~64 kD. We then reconstituted the purified receptor into preformed SUVs via detergent solubilization followed by dialysis, in order to quantify the concentration of the receptor successfully reconstituted into SUV's. We synthesized a highly fluorescent Ni-NTA (R6G-Ni-NTA) label for conjugation to the His-tagged protein. We then exposed the protein containing liposomes to the fluorescent label and subjected them to dialysis to remove excess R6G-Ni-NTA. We then determined the concentration by ultraviolet-visible (UV-VIS) spectrometry using a standard curve based on the absorption spectrum of known concentrations of Rhodamine 6G. From these data, we determined the final quantity of protein to be 260 pM per gram of cells harvested.

(3) Synthesis of highly florescent selective and non-selective 5HT3R agonists for single molecule studies:

A series of novel highly fluorescent selective and non-selective serotonin receptor agonists synthesized for the proposed single molecule experiments.

(4) Optimization of fluorescence assays of protein function in a bilayer coated bead platform:

Protein functionality was assayed by observing the response of reconstituted bacteriorhodopsin after time dependent exposure to broad spectrum visible light. To do this, we loaded porous silica beads with the pH sensitive dye, SNARF-1, prior to bilayer formation. The fluorescence monitored after-exposure to 500–675 nm light using an excitation wavelength of 488 nm. A shift in fluorescence emission due to a pH change after illumination from a xenon lamp indicates proton pumping activity. In order to determine the diffusional behavior of bacteriorhodopsin in this hybrid system, we performed a FRAP experiment using rhodamine-labeled protein. The diffusion constant obtained for the protein from this assay suggests bacteriorhodopsin experiences near native diffusion in bilayers supported by a highly porous silica substrate.

(5) Demonstration of bilayer-coated bead translocation and electrochemical seal using patch-clamp assembly:

We used pulled glass pipettes interfaced to the Axopatch 200B patch-clamp headstage to translocate bilayer coated porous silica beads and obtain gigaohm seals for future high-resolution electrochemical measurements of proteins reconstituted into this bead-based platform.

# Capture and Utilization of Prosody in Disambiguating Spoken Speech

*86801*
*T. L. Bauer*

## Abstract

The Next Generation Intelligent Systems Grand Challenge LDRD developed a unique cognitive framework capability that has been demonstrated in multiple subject domains. That technology now provides the basis for another significant advancement in methods to auto-populate the model with detailed data from specific individuals using speech. This proposed development will significantly expand the applicability of the capability into unstructured domains like knowledge preservation or intelligence analysis using spoken data sources. However, the current state of the technology simply converts speech to text and ignores important cues that could be used to improve the quality of the model building.

We propose to continue investigating methods of using prosodic cues to improve automated analysis of speech beyond what can be accomplished through speech-to-text analysis alone. Prosodic features will be captured and analyzed using audio recordings of speech and software that track the fundamental frequency, harmonics, and amplitude of the speaker's voice. 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. The work will be done in conjunction with the University of Illinois at Urbana-Champaign.

This work builds on research provided in the first year that suggests that utilizing prosodic cues in our model building could have a substantial impact on national security applications involving building models of individuals from their speech. Having determined the most important prosodic cues, we will evaluate how to utilize this information in enhancing our automated model building technologies. Depending on the prosodic features identified, it may be possible to use this information to determine which sections of some spoken speech are least salient for the person and should not be used in the model building.

## Accomplishments

We performed experiments using equi-bias sentences and early/late closure sentences, and analyzed their correlations to event-related potentials in the brain. For the equi-bias items, it appears that the sentences with plausible direct objects elicited an N400 (a component of event-related potentials) at the point of disambiguation. This fits with the prediction that readers would interpret a plausible noun as a direct object of the main verb. For the early/late closure sentences, we did not observe the predicted P600 component (another component of event-related potentials). Instead, there may be a larger N400 in response to the disambiguating word in the early closure sentences. This indicates that the participants are not interpreting these sentences in the way that we predicted. This work provided us enough information to start formulating plans for extending existing Sandia developed technology for building models of individuals from text that they produce.

# Fundamentals of Embossing Nanoimprint Lithography in Polymer Substrates

*93361*

*B. A. Simmons*

## Abstract

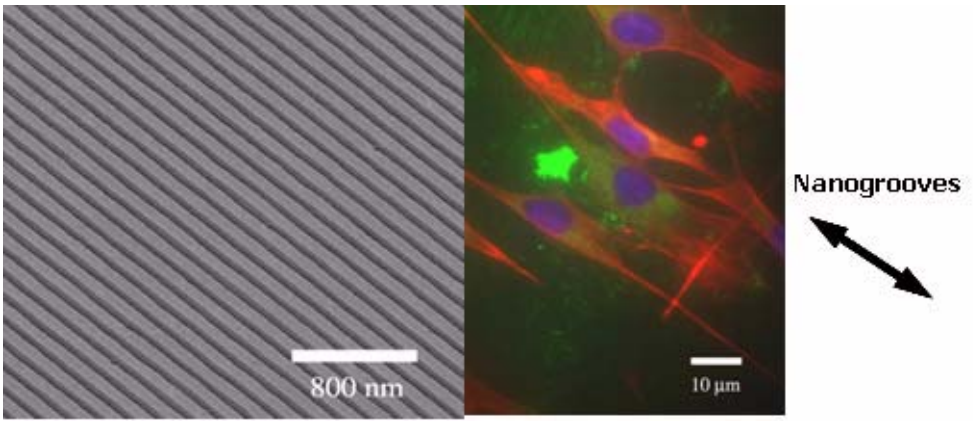
Nanoimprint lithography (NIL) is a nanomanufacturing technique that uses a nanostructured master template to form features in a substrate with best resolution near 1 nm and areas as large as 100 $cm^2$. In embossing-based NIL, a nanostructured master is heated and pressed into a thermoplastic polymer film, forming a negative relief replica in the polymer film. Nearly all previous research on NIL focused on nanoelectronics fabrication in which the polymer film acts as a mask layer. The proposed work seeks to develop the embossed, nanostructured polymer film as the functional surface rather than as a mask layer.

The goal of the proposed research is to develop a first principles understanding of polymer transport during printing so that any polymeric material could be employed for embossing based NIL. The need for this research is particularly acute when polymer nanostructure size is comparable to the polymer molecule radius of gyration, and when the nano scale heat and mass transport properties of the polymer are not known. The lack of fundamental materials science knowledge is the major limit to the 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 material between properties and processing parameters will enable rational NIL process design.

## Accomplishments

The scope of this activity is part of a collaboration between Dr. Bill King of the Georgia Institute of Technology and researchers at Sandia. This activity investigates nanoscale polymer heat and mass transport during embossing based NIL and direct-write atomic force microscope (AFM) nanoindentation. We will manufacture master templates with features in the range of 10 nm–2 micron in silicon and electroplate them into metal to accommodate many embossing trials. A number of thermoplastic polymers will be investigated, with molecular weights in the range 104–107 kDa, corresponding to characteristic molecular length-scales in the range 3–50 nm. We will use AFM to characterize the embossed substrates and correlate embossing conditions, polymer molecular properties, and manufactured feature sizes. Continuum and sub-continuum models of polymer transport and template deformation will aid in this analysis. The ultimate goal is to establish design rules for the manufacture of polymer nano/microdevices.

In addition to developing these design rules for NIL, this project will also investigate 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. We will use the results obtained from this research to develop next generation tissue scaffolds and mitigation of biofouling.




*Images taken of* (a) *nanopatterned polymer substrate and* (b) *patterned cell growth that is controlled by the nanoscale features present.*

## Refereed

J.L. Charest, M.T. Eliason, A.J. Garcia, A.A. Talin, B.A. Simmons, and W.P. King, "Polymer Cell Culture Substrates with Combined Nanotopographical Patterns and Micropatterned Chemical Domains," to be published in *Journal of Vacuum Science and Technology B.*

# Rational Understanding and Control of the Magnetic Behavior of Nanoparticles

*93362*

*B. A. Simmons*

## Abstract

Magnetic spinel ferrite nanoparticles, $MFe_2O_4$ (M = Mn, Mg, Zn, Co, Fe, etc.), have great potential for biomedical diagnostics and targeted drug delivery. To realize biomedical applications of nanoparticles, we must optimize the magnetic properties of the nanoparticles and modify the surface of the nanoparticles 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 hypothesized 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 propose to study the correlations between the coercivity of nanoparticles, the quantum couplings in surface metal atoms, and the surface modification.

## Accomplishments

A better understanding of the fundamental properties of magnetic nanoparticles such as superparamagnetism is crucial to potential applications, including high density information storage, ferrofluid technology, magnetically guided drug delivery, and magnetic resonance imaging (MRI) enhancement. Over the past few years, we systematically studied the effects of size and magnetocrystalline anisotropy (K) upon the superparamagnetic properties of nanoparticles. In addition to crystalline anisotropy, the particle-particle magnetic interactions could contribute to magnetic anisotropy, as well, and consequently change the magnetic properties of nanoparticles. Understanding the interparticle interactions is of fundamental interest and is also vital to certain applications of magnetic nanoparticles such as high density data storage and ferrofluids. Interparticle interactions may arise from dipole-dipole interactions between nanoparticles or the exchange interactions between the magnetic ions at the surface of neighboring particles.

We investigated the effects of interparticle interactions upon the magnetic properties of $CoFe_2O_4$ and $MnFe_2O_4$ nanoparticles via the systematic dilution of nanoparticles in eicosane. We found the blocking temperature for both spinel ferrite systems decreased with dilution, i.e., decreased interparticle interactions. This finding is consistent with Shtrikman-Wohlfarth (SW) and Dormann-Bessais-Fiorani (DBF) models, which conclude that interparticle interactions lead to an increase in the anisotropy energy barrier for magnetization reversal. The blocking temperature for $MnFe_2O_4$ nanoparticles decreased steadily with dilution. The blocking temperature for $CoFe_2O_4$ nanoparticles remained constant until ~15 w/w% concentration, at which point it decreased with dilution. This finding suggests the formation of particle clusters in the strongly interacting $CoFe_2O_4$ system. The reduced remanence (the squareness ratio of magnetic remanence/magnetic saturation) decreased with increasing interactions for $MnFe_2O_4$ nanoparticles, while it increased with increasing interactions for $CoFe_2O_4$ nanoparticles. The increase in reduced remanence for $CoFe_2O_4$ nanoparticles is also consistent with the formation of nanoparticle clusters.

## Refereed

C.R. Vestal, Q. Song, and Z.J. Zhang, "Effects of Interparticle Interactions upon the Magnetic Properties of $CoFe_2O_4$ and $MnFe_2O_4$ Nanocrystals, "*Journal of Physical Chemistry B*, vol. 108, p. 18222, 2004.

# MEMS Dual Backplate Capacitive Microphone

*93364*
*W. K. Schubert*

## Abstract

The objective of this project was the investigation of compliant membranes for the development of a microelectromechanical system (MEMS) microphone using the Sandia Ultra-planar, Multi-level, MEMS Technology 5 (SUMMiT V™) fabrication process. The microphone is a dual backplate capacitive microphone that uses electrostatic force feedback. The microphone consists of a diaphragm and two porous backplates, one on either side of the diaphragm. This forms a capacitor between the diaphragm and each backplate. As the incident pressure deflects the diaphragm, the value of each capacitor will change, thus resulting in an electrical output. Feedback may be used in this device by applying a voltage between the diaphragm and the backplates. This voltage creates an electrostatic force that balances the incident pressure keeping the diaphragm stationary.

We finished fabrication of the MEMS capacitive microphone and post-processed it at the University of Florida. We packaged the microphone and completed initial testing. The packaged microphone demonstrates a flat frequency response, a linear response up to the designed limit, and a sensitivity that is close to the designed value. Future work will focus on characterization of additional devices, extending the frequency response measurements, and investigating the use of other types of interface circuitry. Sandia provided access to, and training for, the SUMMiT V process.

## Accomplishments

We used the SUMMiT V process at Sandia to fabricate the structural layers of the microphone. This process mainly consists of the deposition and patterning of alternating layers of polysilicon and silicon dioxide. Polysilicon is used to form the microphone structure, and the silicon dioxide is a temporary, or sacrificial, material to support the various layers of polysilicon during fabrication. After completion of the SUMMiT V process, we performed a series of post processing steps to release the device and complete the fabrication.

The process flow for the dual backplate capacitive microphone begins with a 6 inch silicon wafer. We then deposit a layer of silicon dioxide and silicon nitride. These insulate the polysilicon structure from the silicon substrate. In addition, the silicon nitride provides good adhesion for the polysilicon layers. We then deposit the first layer of polysilicon, poly0. This polysilicon layer forms a base for the anchors and for electrical interconnections. A 2 µm layer of sacrificial oxide forms a spacer between the poly0 electrical connections and the next layer of polysilicon. We form the bottom backplate by depositing and patterning poly2. The bottom air gap space is held by the next sacrificial layer, sacox3. The diaphragm is then formed from poly3. The final steps of the SUMMiT V process create the top backplate.

The microphone returned from Sandia in the form of unreleased die. We conducted the remainder of the processing at the University of Florida. To facilitate processing on individual die, we designed a 4 inch handle wafer to hold the microphone die. The handle wafer consists of a silicon wafer joined to a Pyrex wafer. We etched a cavity in the silicon wafer to hold the microphone die. Pyrex is used for the lower layer because this layer must be transparent for front to back alignment. The first step of the post processing is to remove layers of oxide and polysilicon from the backside of the microphone chip. We accomplished this via mechanical lapping. Then we etch the silicon substrate below the microphone structure using a deep reactive ion etch. This is followed by two etches to remove the thin layers of oxide and nitride. Finally, we remove the sacrificial oxide to release the microphone structure. To avoid stiction, we dry the microphone using supercritical $CO_2$. This completes the microphone fabrication.

We designed and fabricated the dual backplate microphone. Preliminary characterization of the device shows that it is well suited for aeroacoustic measurements. The microphone output voltage demonstrated a linear response up to 160 dB. The frequency response of the microphone is flat up to 20 kHz, which was the limit of the experimental setup. We found the resonant frequency to be 230 kHz. The frequency response should extend up to near the resonant frequency. At a frequency of 1 kHz, the equivalent pressure noise is 42 dB/RT Hz. The interface circuitry is the dominant source of low frequency noise. Improvements to the

interface circuitry could lower the noise floor. At 1 kHz, the microphone has a dynamic range of 42 dB to 160 dB for a 1 Hz bin. The microphone specifications are listed below:

| *Specification* | *Value* |
|---|---|
| Dynamic range | |
| at 1 kHz (1 Hz bin) | 60 dB |
| Measured bandwidth | 300 H–20 kHz* |
| Measured resonant frequency | 230 kHz |

* Limited by measurement setup. The bandwidth should extend to the first resonance at 230 kHz.

We packaged the microphone at the University of Florida and completed initial testing. The microphone demonstrates a flat frequency response, a linear response up to the designed limit, and a sensitivity that is close to the designed value. Future work will focus on characterization of additional devices, extending the frequency response measurements, and investigating the use of other types of interface circuitry.

## Other Communications

D.T. Martin, K. Kadirvel, J. Liu, R.M. Fox, M. Sheplak, and T. Nishida, “Surface and Bulk Micromachined Dual Back-Plate Condenser Microphone,” presented and in *Proceedings of the 18th IEEE International Conference on Micro Electro Mechanical Systems*, Miami Beach, FL, January 30, 2005, vol. 1, pp. 319–322.

# Flow Control and Mixing in Microfluidic Devices

*93365*

*A. C. Sun, C. M. Brotherton*

## Abstract

Our research group is studying flow control and mixing in microfluidic devices. We use living radical photopolymerization (LRPP) to produce polymeric microfluidic devices. LRPP allows for excellent control of microchannel geometry and can be used to chemically modify the surfaces of the channels by attaching charged species or other functional groups. For flow control, we focused on manipulating electroosmotic flow by manipulating the zeta potential. Using LRPP, we have surface grafted materials to change the zeta potential of the polymeric material used for construction. Additionally, we controlled the zeta potential of the polymeric surface by manipulating the exposure time for the surface grafting. By selecting the appropriate material and exposure time, we can tailor the zeta potential of the polymeric surfaces for different applications.

For mixing, we investigated multiple techniques for use in microfluidic devices. The first techniques utilize porous polymer plugs. The plugs are made from a mixture of monomer and salt. After polymerization, the salt is dissolved, leaving a porous polymer. The size of the pores in the polymer plug is dictated by the size of the salt crystals used in construction. Using a salt crystal size range of 75–106 microns, the extent of mixing is 3–5 times higher than in a similar channel without a porous polymer plug mixer. The flow rate range investigated was 10 to 80 µL/min.

The other techniques investigated are hydrodynamic focusing and focusing using electroosmosis and physical constrictions. Each of these techniques constricts the mixing fluids and reduces the characteristic diffusion length. We performed simulations to evaluate the important dimensionless groups to consider when designing a microfluidic mixer that utilizes one of these techniques. Typical results show that the extent of mixing increases with increasing constriction. The simulation results allow for improved mixers to be designed.

## Accomplishments

Using LRPP, we investigated modifying the zeta potential of the channel walls and in turn investigated manipulating the electroosmotic flow in the channel. The typical substrate material used for fabrication of the polymeric microfluidic device consists of a mixture of an aromatic urethane diacrylate and triethylene diacrylate. The substrate exhibits a negative zeta potential. We investigated grafting two materials on the surface of the substrate to change the zeta potential. We polymerized the surface grafts using a collimated light source with an intensity of 45 mW/cm$^2$ for 900 seconds. The first material investigated is polyethylene glycol 375 acrylate (PEG375). The PEG375 surface graft exhibits a neutral zeta potential over a pH range of 2–10. We expected this result since PEG375 does not contain any functional groups that significantly accept or donate protons. The second surface graft material is 2-(dimethylamino)ethyl methacrylate (DMAEMA). The DMAEMA surface graft exhibits a positive zeta potential. The positive zeta potential is the result of the amine groups accepting protons from the electrolyte. Additionally, we performed experiments to determine the effect of exposure time on the zeta potential of a surface grafted with DMAEMA. Below 200 seconds, the coverage of DMAEMA is too low to affect the zeta potential. Above 450 seconds, the surface has complete coverage, and the zeta potential is due to the DMAEMA alone. Between 200–450 seconds, the zeta potential of the surface is the result of a contribution from the surface graft and the substrate. By selecting the appropriate material and exposure time, we can tailor the zeta potential of the polymeric surfaces for different applications.

Mixing, by diffusion, is typically a slow process on microfluidic devices. Our research group focused on investigating multiple techniques to reduce the mixing length in microfluidic flows. The first technique uses porous polymer plugs. We fabricate porous polymer plugs using photopolymerization to polymerize a monomer/salt paste. The size of the salt crystals determines the final size of the pores in the porous polymer plugs. After polymerization, we wash the plug with deionized water to dissolve the salt. Experiments focused on porous polymer plugs made with two salt crystal size ranges. The first salt crystal size range is 75–106 microns. For flow rates, the extent of mixing is

between 3 and 5 times better than a similar microfluidic channel without a plug. The extent of mixing is determined by comparing the concentration profile of a solute before and after the porous polymer plug. The second salt crystal size range is 53–126 microns. This salt crystal size range also resulted in an extent of mixing similar to the first salt crystal size range. The heterogeneity of the two salt crystal size ranges was not different enough to affect the extent of mixing. The results will help design future porous polymer plug mixers.

The other mixing techniques investigated are hydrodynamic focusing and focusing using electroosmosis and physical constrictions. Each of these techniques constricts the mixing fluids and reduces the characteristic diffusion length. We simulated the microfluidic channels using these techniques with GOMA software. The simulations allowed us to determine the quantitative effects of important dimensionless groups that affect the extent of mixing. The key dimensionless group is a modified form of the Peclet number. The Peclet number describes a ratio between the characteristic diffusion time and the characteristic convection time. The modified form we use includes two geometry parameters. One parameter describes the height of the channel, and the other describes the length of the mixing region. Typical results show that the greater the constriction of the mixing streams, the more effective the mixer is.

## Refereed

H.M. Simms, B.T. Good, C.M. Brotherton, K. Anseth, R.H. Davis, and C.N. Bowman, “In Situ Fabrication of Macroporous Polymer Networks by Living Radical Photo-Polymerization and Leaching Within Microfluidic Devices,” *Lab-on-a-chip*, vol. 5, pp. 151–157, 2005.

B.T. Good, C.M. Brotherton, D.S. Smirnova, R.H. Davis, and C.N. Bowman, “Integration of Conductive Materials into Polymeric Devices for Portable Microfluidic Applications,” to be published in *Lab-on-a-chip.*

# Process Science and Engineering for Thermomechanical Nanomanufacturing

*93366*

*A. C. Sun*

## Abstract

Nanoimprint lithography (NIL) is a high resolution, large scale, high throughput manufacturing alternative to standard silicon based fabrication technologies. In the nanoimprint embossing process, a nanopatterned silicon master is heated and pressed into a thermoplastic polymer film. Heat and force transfers the master pattern in a negative relief replica in the polymer sample, resulting in feature sizes as small as 10 nm over areas > 1000 $cm^2$. Critical to transition of embossing-based nanomanufacturing from laboratory to commercial use is the ability to predict polymer flow during embossing. In practice, NIL can result in incomplete filling or nonuniform residual layers due to lack of tools for predicting polymer flow. Various phenomenological models of polymer flow have been developed, yet no unified and consistent understanding of process variables determining flow profile or filling time exists. This work identifies critical geometries, materials properties, and process conditions that govern polymer flow during NIL embossing, thus allowing NIL design rules to be formulated.

## Accomplishments

Finite element simulations of polymer flow during NIL examined rectangular cavities embossing a Newtonian fluid under viscous-dominant conditions. Simulations varied mold geometry parameters of indenter width (S), cavity width (W), cavity height (hc), and initial polymer film thickness (hi) for single cavities and non-uniform asymmetric cavities. The continuum simulations captured the NIL physics observed by numerous published articles from the 10 nm to 1 mm scale, showing that polymer deformation and fill time is governed by location and rate of polymer shear during imprinting, exhibiting deformation predominantly close to the vertical indenter sidewall that can result in single peak or dual peak deformation modes.

Polymer deformation mode and cavity filling time can be predicted based on ratios of hc, W, S, and hi. Single peak deformation depends solely on W/hi and always occurs when W/hi < 1. Single peak, dual peak shear, and dual peak squeeze flow follow different fill time dependencies predicted by viscous flow theory of laminar flow between plates, Stoke's flow, and squeeze flow. Squeeze flow occurs when S/hr > 1, where hr is the residual film thickness. By determining the proper flow mode based on mold geometry and polymer film thickness ratios, a characteristic NIL velocity (VNIL) can be identified, allowing establishment of NIL embossing design rules based on NIL Capillary number (CaNIL), directional flow ratio W/hi, and polymer supply ratio S/hr.

The characteristic VNIL predicts the cavity filling order of non-uniform cavities. Individual cavity size, not total filling volume, dominates replication time, with large differences in individual cavity size resulting in nonuniform, squeeze flow filling. Maximum indenter width governs filling time with a quadratic dependence, and the rapid filling of small cavities results in an increase in effective maximum indenter width. High density features can be modeled as a solid indenter in squeeze flow to accurately predict polymer flow and allow for optimization of wafer-scale replication.

The present simulations make it possible to design imprint templates capable of distributing pressure evenly across the mold surface, and to facilitate symmetric polymer flow over large areas to prevent mold deformation and non-uniform residual layer thickness. Three parameters, W/hi, S/hr, and CaNIL, accurately predict polymer deformation and filling mode. The directional flow ratio W/hi predicts the importance of deviatoric stress determining single or dual peak deformation and, hence, vertical or lateral flow. The polymer supply ratio S/hr predicts the influence of squeeze flow and relative filling times independent of material rheological properties. The CaNIL predicts the flow driving mechanism to distinguish the necessary process design from the external embossing setup and internal material physics. The three parameters W/hi, S/hr, and CaNIL combine to allow a rational and predictive NIL process design and provide a step towards establishing design rules for NIL. Rather than rely on improvised trial and error, it is possible to use these tools to appropriately tailor tool geometry, process conditions, and

polymer rheology and surface chemistry for optimal NIL over a predicted timescale.

## Refereed

H.D. Rowland, W.P. King, A.C. Sun, and P.R. Schunk, “Impact of Polymer Film Thickness and Cavity Size on Polymer Flow During Embossing: Towards Process Design Rules for Nanoimprint Lithography,” presented at the 49th International Conference on Electron, Ion, and Photon Beam Technology and Nanofabrication Conference, Orlando, FL, 2005.

H.D. Rowland, W.P. King, A.C. Sun, and P.R. Schunk, “Simulations of Non-Uniform Embossing: The Effect of Asymmetric Neighbor Cavities on Polymer Flow During Nanoimprint Lithography,” submitted to *Journal of Vacuum Science and Technology B.*

H.D. Rowland, W.P. King, A.C. Sun, and P.R. Schunk, “Impact of Polymer Film Thickness and Cavity Size on Polymer Flow During Embossing: Towards Process Design Rules for Nanoimprint Lithography,” submitted to *Journal of Micromechanics and Microengineering.*

# Micromechanics Modeling of Multifunctional Nanocomposites

*93367*
*D. C. Hammerand*

## Abstract

Recent efforts at manufacturing composites consisting of carbon nanotubes (CNTs) embedded in a polymer matrix resulted in what can be termed multifunctional nanocomposites. In such composites, the CNTs serve to increase the stiffness of the overall composite by appreciable amounts at volume fractions less that 5% (30% increase in Young's modulus with 1% volume fraction of CNTs). The electrical properties of CNTs, which can be either metallic or semi conducting depending on their chirality, lead to composites in which small volume fractions of metallic CNTs can cause large increases in composite conductivity. Similar results are expected for thermal conductivity. Likewise, equally small amounts of semi-conducting CNTs have a strong effect on the dielectric properties of the composite. Perhaps even more significant, evidence suggests that under certain deformations, metallic CNTs can become semiconducting, thus indicating electromechanical coupling. The goal of the present work is to use micromechanics approaches for finding effective composite mechanical, thermal, and electrical properties.

We applied micromechanics techniques for obtaining the effective elastic constants for CNT reinforced composites that account for CNT bundling, the presence of interphase into the calculation of the effective elastic properties regions due to functionalization and/or polymer wrapping, and random orientations of CNT/CNT bundles. We incorporated the use of cohesive zones to account for short range interactions across the interface between the CNTs and the surrounding interphases. We also incorporated the use of functionally graded material properties to more accurately represent interphase behavior. Similarly, we will incorporate the effects of aligned versus random orientation and the presence of interphase regions incorporated into the micromechanics predictions for effective thermal conductivity, effective thermal expansion, effective electrical conductivity, and effective dielectric constant. Finally, an attempt will be made to use micromechanics to address electrothermomechanical coupling in CNT reinforced composites.

## Accomplishments

The third year of this project focused on the further development of the single-step composite cylinders approach to the modeling of CNT reinforced composites. The previous approach was directly correlated with an energy equivalence approach, which leads to the development of a generalized self-consistent composite cylinders (GSC-CC) approach. In the GSC-CC approach, we embedded the previous composite cylinders arrangement in the effective material. This resulted in a coupled set of nonlinear algebraic relationships between the effective properties of the composite and allowed the interactions between fibers to be accounted for in the effective material as opposed to the matrix phase. We solved for the effective properties numerically and found them to be identical to the previous single-step composite cylinders approach, further validating the previous approach.

We also conducted efforts toward modeling an interphase region with graded material properties corresponding to the variation in properties. This is hypothesized to exist in surrounding nonfunctionalized and functionalized CNTs due to CNT interaction with the surrounding polymer. We used a series of concentric interphases to represent the graded interphase with subregions of piecewise constant material properties that necessitated the implementation of the n-phase composite cylinders approach. We employed the n-phase composite cylinders approach to model the graded interphase in polyethylene nanocomposites observed through density variations in molecular dynamics simulations available in the literature. We conducted efforts at modeling the same interphase with a single interphase represented with continuous nonconstant functions, but we have not been successful to date.

Finally, returning to a two-step approach, we used the n-phase composite cylinders solution to generate concentration factors for a Mori-Tanaka method that we then employed to model the effects of random orientation of CNT reinforced composites. We also made attempts at obtaining the concentration factors directly from the composite cylinders solution.

## Other Communications

G.D. Seidel and D.C. Lagoudas, "Modeling of Carbon Nanotube Composites," presented at the NASA URETI Workshop, College Park, MD, October 2004.

G.D. Seidel, D.C. Lagoudas, S.J.V. Frankland, and T.S. Gates, "Modeling Functionally Graded Interphase Regions in Carbon Nanotube Reinforced Composites," presented and in *Proceedings for the 20th American Society for Composites Technical Conference*, Philadelphia, PA, September 2005, CD-ROM.

G.D. Seidel, D.C. Lagoudas, and D.C. Hammerand, "Micromechanical Analysis of Clustering and Load Transfer in Carbon Nanotube Composites," presented and in *Proceedings of the 2005 Joint ASME/ASCE/SES Conference on Mechanics and Material*, Baton Rouge, LA, June 2005, CD-ROM.

D.C. Hammerand, G.D. Seidel, and D.C. Lagoudas, "Computational Micromechanical Analysis of Single-Walled Carbon Nanotube Reinforced Composites," submitted to *Composites Science and Technology.*

D.C. Hammerand, G.D. Seidel, and D.C. Lagoudas, "Finite Element Micromechanical Analysis of Clustering and Load Transfer in Carbon Nanotube Composites," presented and in *Proceedings of the 8th U.S. National Congress on Computational Mechanics*, Austin, TX, July 2005, CD-ROM.

# Fabrication and Device Applications of Aligned Mesoporous Architectures

*93369*

*C. J. Brinker*

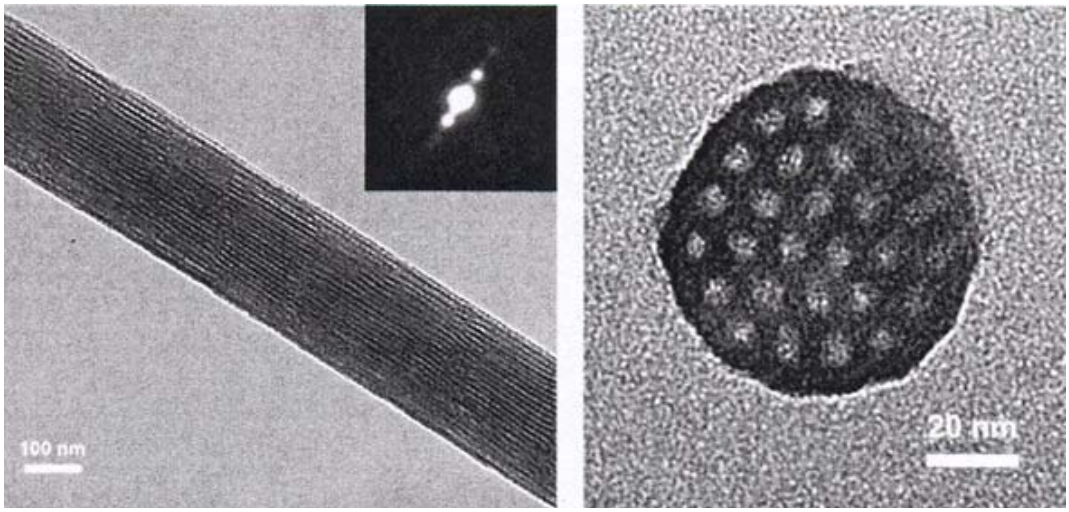

## Abstract

This project was awarded in July 2005 to Professor Yunfeng Lu, Tulane University, as the recipient of the Department of Energy (DOE) Presidential Early Career Award for Scientists and Engineers. During the last decade, mesoporous materials with tunable periodic pores have been synthesized using surfactant liquid crystalline fluids as templates, opening a new avenue for a wide spectrum of applications. However, their applications are somewhat limited by their unfavorable pore orientation. Although a great effort has been devoted to align the pore channels, fabrication of mesoporous materials with perpendicular pore channels remains challenging. This collaborative project between Tulane University and Sandia National Laboratories will investigate fabrication of mesoporous materials with perpendicularly aligned pore channels.

We hypothesize that the mesoporous channels can be aligned using capillary forces. Conceptually, by contacting a lyotropic liquid crystalline fluid that contains inorganic or organic building blocks (e.g., silicate) and surfactant molecules with anodized porous alumina membranes, the capillary force generated by the curvature of the fluid meniscus causes capillary rise of the complex fluid, gradually filling the alumina pore channels with aligned liquid crystalline templates. Subsequent removal of the templates creates mesoporous silica membranes with perpendicular pore channels.

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, such 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.

We performed initial investigations of the capillary growth of hexagonal mesostructured silica/copolymer composites within the cylindrical pores of an anodized alumina disk. We put a freestanding hydrophilic porous alumina membrane onto a liquid crystalline hexagonal silicate/copolymer composite film with appropriate pressure. This resulted in the filling of cylindrical alumina pores with the liquid crystalline silicate/surfactant mesophase—a process driven by capillary force. The preliminary results show that the shearing force induced by interfacial capillary force aligns the hexagonal tubules of silicate/copolymer mesophase along the direction of alumina pore channels. After removal of the templating copolymers by calcination, we generated mesoporous silica nanowires with desirable oriented mesopore channels perpendicular to the surface of the alumina disk.


*Side-view and cross section TEM images of a single oriented mesoporous silica nanowire that shows hexagonal organized mesophase.*

## Accomplishments

We performed a preliminary demonstration that mesoporous channels can be aligned using capillary forces. By contacting a liquid crystalline hexagonal silicate/copolymer composite film with porous anodized alumina membranes, the liquid crystalline silicate/surfactant mesophase is gradually filled into the cylindrical alumina pores driven by the capillary force. The side view and cross section transmission electron microscope (TEM) images clearly reveal the mesopore channels are predominantly oriented along the long axes of the silica nanowires, i.e., the silica nanopore channels are perpendicular to the surface of alumina disks. The two-dimensional (2-D) hexagonal arrangement of the mesophase remained during the alignment. It was found that the thermal treatment (e.g., heating at a specific temperature) is of importance for the alignment of pore channels, providing sufficient mobility of the mesophase. We investigated various surfactants and conditions for the formation of tunable mesostructures under the capillary forces. For example, we prepared oriented hexagonal mesoporous silica nanowires using copolymer F127 as structural directing agent. We also prepared concentric lamellar and cubic mesostructured silica nanowires using P123 as structural directing agents by changing the synthetic conditions. Future work will emphasize the synthesis of oriented mesoporous silica nanowires using alumina membranes with varying diameters ranging from 30 nm to 250 nm.

# Characterization of Nanomagnetic Materials

*93736*
*M. P. de Boer*

## Abstract

Molecular nanomagnets are a relatively new class of materials that exhibit high-spin magnetic moments. Micro- and nanodevices that could be enabled by nanomagnets will require a reproducible process for creating thin films. The instability of these nanomagnets at high temperatures precludes the use of many established thermal techniques for depositing thin films. Recent work on the development of non-thermal laser techniques for the deposition of thin films of the molecular magnet, manganese12-acetate (Mn12), led to the ability to create quality films of molecular magnets. Previous results in work on patterning these films allow studies of hybrid materials made of a layer of nanomagnets in close proximity to metallic, semiconducting, or superconducting layers. These studies could generate greater understanding of the effects of localized magnetic fields on electron transport in metals and have possible applications in quantum computing and magnetic storage.

## Accomplishments

We performed laser ablation with a nitrogen laser and an excimer laser to create the thin films. We carried out characterization of the films using x-ray photoelectron spectroscopy, electron-spray ionization mass spectrometry, atomic force microscopy, and superconducting quantum interference device magnetometry. Evidence that ablation with the nitrogen laser or excimer laser at low energies produces films maintaining the important magnetic and molecular properties diminished the need for further exploration of the previously proposed matrix assisted pulsed laser evaporation and matrix assisted femtosecond laser evaporation techniques. Two papers were accepted for publication in refereed journals. We submitted another paper for publication in the *Proceedings Journal of the 24$^{th}$ International Conference on Low Temperature Physics* (refereed). We presented a poster concerning this work at the same conference in Orlando, FL, as well as the Conference on Molecular Magnets and Magnetic Hybrid Nanostructures held at the International Center for Theoretical Physics in Trieste, Italy. We also spent much time in the past year working to get our dilution refrigerator into operation so that the next phase of our research can go forward. The refrigerator is now operational.

## Refereed

J. Means, R. Srivastava, V. Meenakshi, W. Teizer, H. Zhao, K. Dunbar, A.A. Kolomenskii, and H.A. Schuessler, “Films of Molecular Magnets Deposited by Low Energy Laser Ablation,” *Journal of Magnetism and Magnetic Materials*, vol. 284, p. 215.

K. Kim, D. Seo, J. Means, V. Meenakshi, W. Teizer, H. Zhao, and K. Dunbar, “Lithographic Patterns of Molecular Magnets,” *Applied Physics Letters*, vol. 85, p. 3872, 2004.

J. Means, R. Srivastava, V. Meenakshi, W. Teizer, A.A. Kolomenskii, H.A. Schuessler, H. Zhao, and K. Dunbar, “Production and Applications of Thin Films of Manganese-12 Molecular Magnets,” to be published in the *Proceedings of the 24th International Conference on Low Temperature Physics*.

R.E. Schaak, A.K. Sra, B.M. Leonard, R.E. Cable, J.C. Bauer, Y.-F. Han, J. Means, W. Teizer, Y. Vasquez, and E.S. Funck, “Metallurgy in a Beaker: Nanoparticle Toolkit for the Rapid Low-Temperature Solution Synthesis of Functional Multimetallic Solid-State Materials,” *Journal of the American Chemical Society*, vol. 127, p. 3506, 2005.

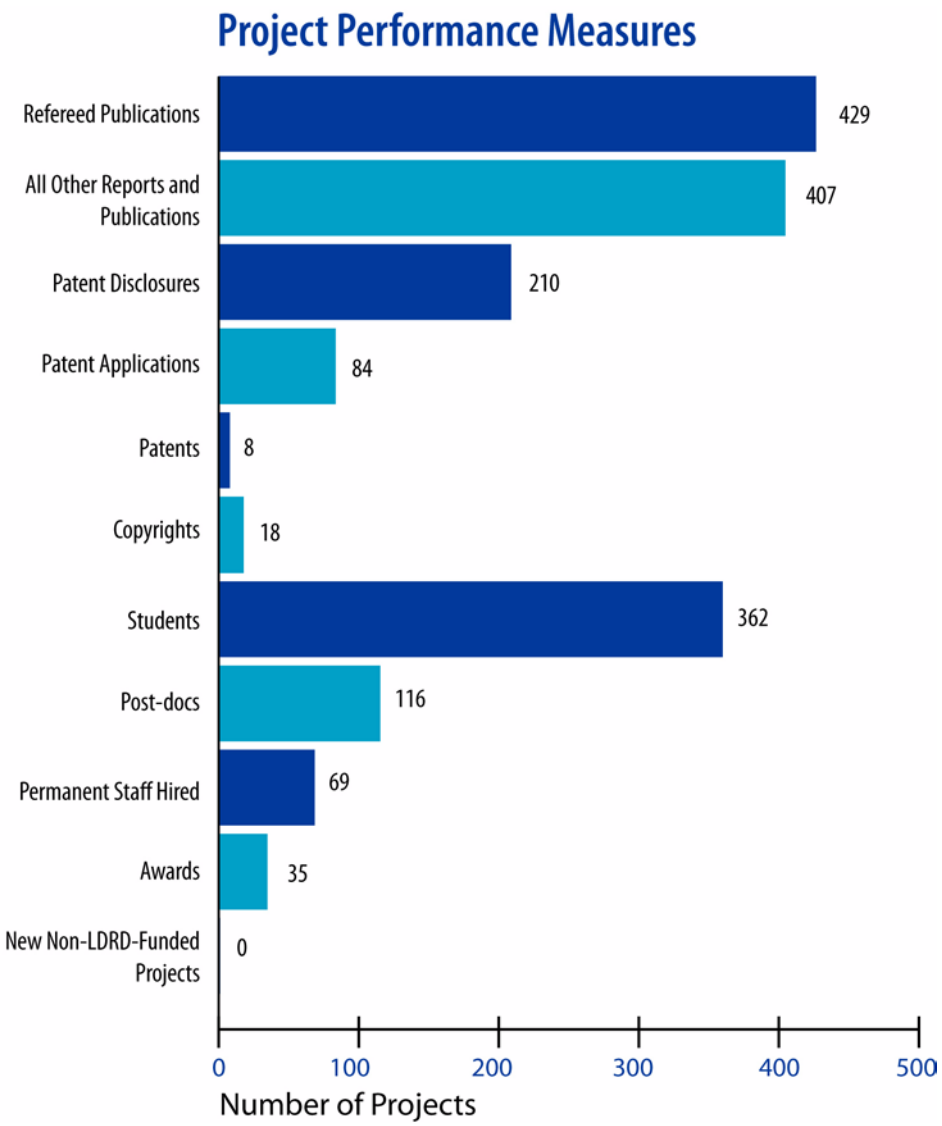
## Appendix A: Awards/Recognition List

| *Awards/Recognition* | *Project Number* | *Project Title* |
|---|---|---|
| Analyzing the Genetic Basis of Metabolic Latency/Reactivation NIH/National Heart, Lung, and Blood Institute K25 Quantitative Career Development Award | **52541** | A Parallel Circuit Simulator for Cell Biology |
| First prize for "Science as Art" at the Materials Research Society Spring Meeting, March 28–April 1, 2005 San Francisco, CA | **67079** | Nanolithography Directed Materials Growth and Self-Assembly |
| 2005 EAS Award for Achievements in Chemometrics | **67081** | 3-D Optical Sectioning with a New Hyperspectral Deconvolution Fluorescence Imaging System |
| Best poster award in physical category for "Solute Distribution in Electrodeposited Ni-Mn Alloys by Atom Probe Tomography" at the Microscopy and Microanalysis Meeting, Honolulu, HI, 2005 | **67082** | The Science of Solutes: Transition Metals in LIGA Nickel |
| 2005 University of New Mexico Senior Faculty Research Excellence Award: C. J. Brinker | **73185** | Superhydrophobic Surface Coatings for Microfluidics and MEMS |
| 2005 Student Internship Program Outstanding Technical Manager/Adviser: Malcolm Carroll | **79844** | Single Photon Sensitive Imaging Detector Arrays |
| Sandia National Laboratories/Stanford University Excellence in Engineering Assistantship | **79893** | Cohesive Zone Modeling of Failure in Geomaterials: Formulation and Implementation of a Strong Discontinuity Model Incorporating the Effect of Slip Speed on Frictional Resistance |

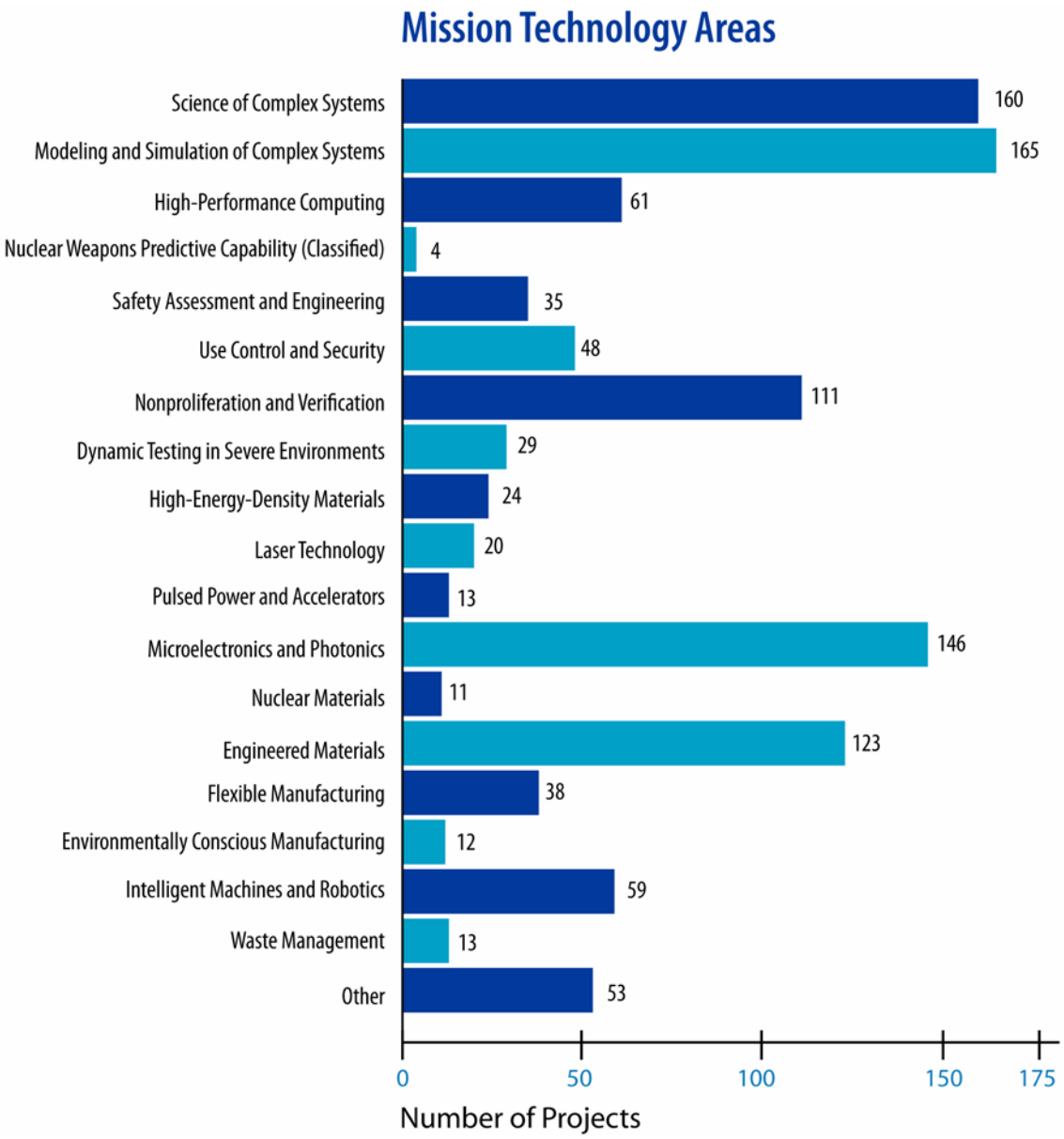
## Appendix A: Awards/Recognition List

| *Awards/Recognition* | *Project Number* | *Project Title* |
|---|---|---|
| Washington State University President's Leadership Award: Marian S. Kennedy | **80595** | Reliability of Materials in MEMS: Residual Stress and Adhesion in a Micro Power Generation System |
| – New Faculty Research Award given by the SE Section of ASEE<br><br>– Two student awards at the regional AlChE meeting and the sectional ASEE meeting | **82854** | Developing Novel Scaffolds for Biological Molecules by Solving the I-QSAR Problem Using the Signature Molecular Descriptor |
| 2005 DOE Presidential Early Career Award for Scientists and Engineers: Yunfeng Lu | **93369** | Fabrication and Device Applications of Aligned Mesoporous Architectures |

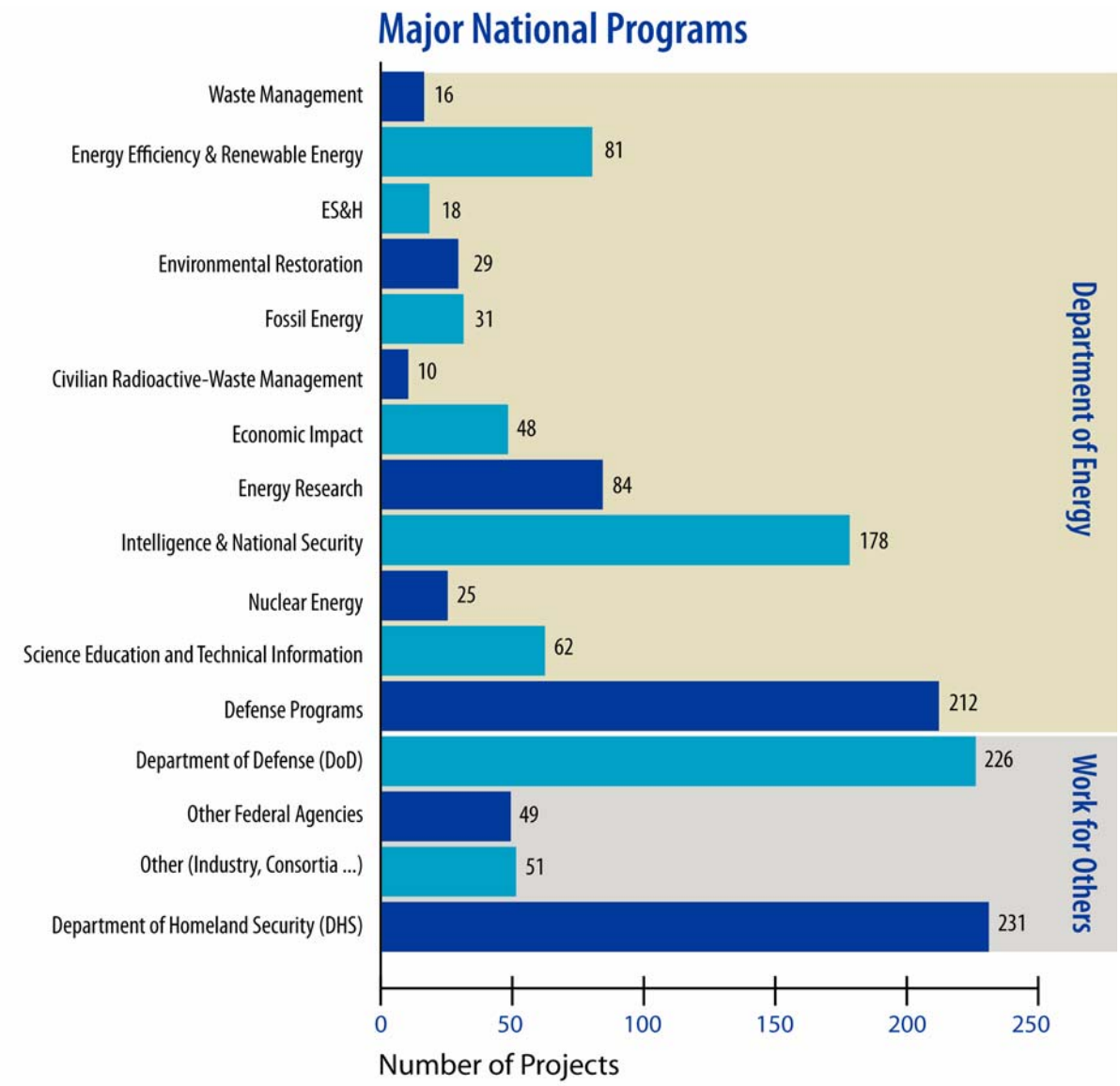
## *Appendix B: Project Performance Measures*



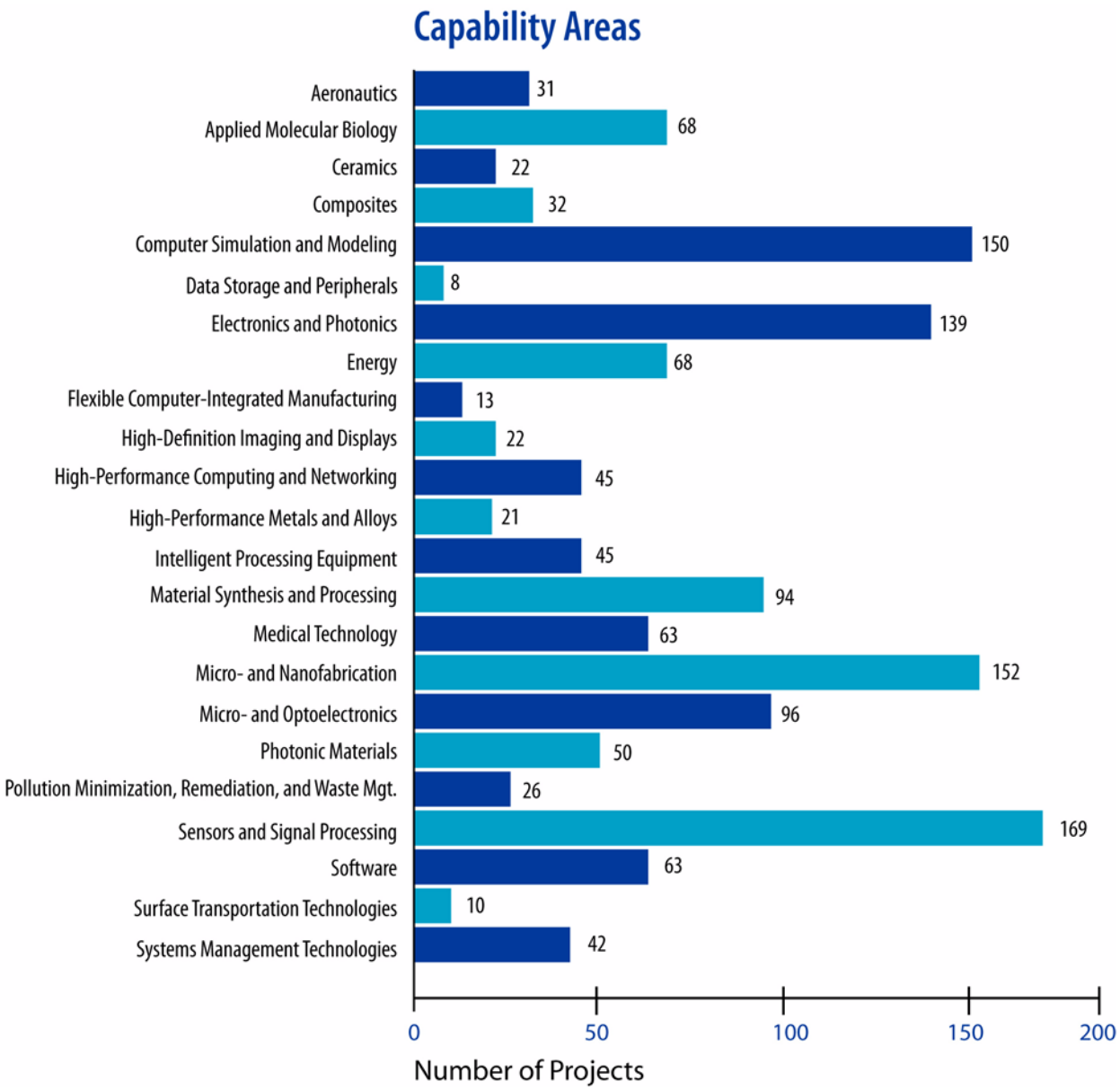
## *Appendix C: Mission Technology Areas*



## *Appendix D: Major National Programs*



## *Appendix E: Dual Benefit Areas and Single-Use Categories*



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