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A Review of Research in the Field of Nanorobotics

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Abstract

This report highlights the findings of an extensive review of the literature in the area of nanorobotics. The main goal of this midyear LDRD effort is to survey and identify accomplishments and advancements that have been made in this relatively new and emerging field. As a result, it may be determined what routes in the area of nanorobotics are scientifically plausible and technically useful so that the Intelligent Systems and Robotics Center can position itself to play a role in the future development of nanotechnology.

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Contents

1. INTRODUCTION	7
1.1 Nanotechnology	7
1.2 Nanorobotics	8
1.3 Organization of the Report	9
2. BIOLOGICALLY INSPIRED NANOROBOTS.....	11
2.1 Biological Molecular Machines	11
2.1.1 Myosin, Kinesin, and Dynein Motor Proteins	12
2.1.2 Flagella Motors	12
2.2 Theoretical Design, Control, and Simulation	13
3. ROBOTIC NANOMANIPULATION	15
3.1 Electron Microscopy	15
3.1.1 Scanning Electron Microscope (SEM)	15
3.1.2 Transmission Electron Microscope (TEM)	16
3.2 Scanning Probe Microscopy (SPM).....	16
3.2.1 Scanning Tunneling Microscope (STM).....	16
3.2.2 Atomic Force Microscope (AFM).....	16
3.3 Nanomanipulation with the SPM	18
3.4 Manipulation of Atoms.....	19
3.5 Manipulation of Nanoparticles	19
3.6 Manipulation of Carbon Nanotubes.....	21
3.7 Manipulation of Biological Materials	22
3.8 Nanoscale Gripping.....	24
3.8.1 Optical Tweezers	24
3.8.2 Nanotweezers	24
3.8.3 Dielectrophoresis	26
3.9 Joining Nanostructures.....	27
3.9.1 Nanosoldering.....	27
3.9.2 Nanowelding	27
3.9.3 Gluing	28
3.9.4 Sintering.....	28
3.9.5 Chemical Bonding.....	28
3.10 Cutting Nanostructures.....	29
3.10.1 Mechanical Cutting	29
3.10.2 Electron Ablation.....	29
4. THE ZYVEX NANOMANIPULATION SYSTEM	31
4.1 The Zyvex S100 Nanomanipulator	31
4.2 Preliminary Testing.....	34

5. CONCLUSION	37
5.1 Summary	37
5.2 Recommendations for Future Work.....	38
6. REFERENCES	39

Figures

Figure 4.1. The Zyvex Nanomanipulation System	31
Figure 4.2. Zyvex S100 Mounted on SEM Stage	32
Figure 4.3. Zyvex S100 Controller Cabinet	33
Figure 4.4. Zyvex Patch Panel Rack.....	34
Figure 4.5. Nickel Particle Lifted by Zyvex Probe.....	35
Figure 4.6. Particle Attached to Probe Using DEP	36

1. Introduction

“...people tell me about miniaturization, and how far it has progressed today... but that’s nothing; that’s the most primitive, halting step in the direction I intend to discuss. It is a staggeringly small world that is below. In the year 2000, when they look back at this age, they will wonder why it was not until the year 1960 that anybody began to seriously move in this direction.” - Feynman, 1959

1.1 Nanotechnology

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” (Feynman, 1959). 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 has been defined as a description of activities at the level of atoms and molecules that have applications in the real world (Ummat et al., 2004a; 2004b). Nanotechnology comprises technological developments on the nanometer scale, usually on the order of 0.1 to 100 nm. A nanometer is one billionth of a meter ($1 \text{ nm} = 10^{-9} \text{ m}$). For a perspective of this scale at the atomic level, a hydrogen atom’s diameter is on the order of an Ångström ($1 \text{ Å} = 0.1 \text{ nm}$). Thus, ten hydrogen atoms laid side by side would measure a distance of about 1 nm across. Nanotechnology is necessarily a multidisciplinary field which encompasses and draws from the knowledge of several diverse technological fields of study including chemistry, physics, molecular biology, material science, computer science, and engineering (Drexler, 1992; Requicha, 2003).

Advances in the field of nanotechnology have expanded the breadth of potential applications tremendously in recent years. The nanotechnology research and development (R&D) areas have been growing rapidly throughout the world. The nanotechnology R&D investment reported by government organizations around the world has increased from approximately \$432 million in 1997 to about \$3 billion in 2003 alone (Roco, 2003). At least 30 countries have initiated national activities in this field (Roco, 2001). Although nanotechnology is currently still considered to be in the precompetitive stage, the worldwide annual industrial production in the nanotechnology sectors is estimated to exceed \$1 trillion in 10 to 15 years, which would require about 2 million nanotechnology workers (Roco and Bainbridge, 2001).

Although its applied use is still limited, nanotechnology has already begun to appear in various applications and products, namely nanomaterials. According to information provided by the National Nanotechnology Initiative (NNI) website, nanomaterials are being used in a number of industries to improve product functionality for electronic, magnetic, optoelectronic, biomedical, pharmaceutical, cosmetic, energy, catalytic, and materials applications. In addition, it has been reported that the areas currently producing the greatest revenue are the use of nanoparticles for chemical-mechanical polishing,

magnetic recording tapes, sunscreens, automotive catalysts, biolabeling, electroconductive coatings, and optical fibers. Although still considered to be in its infancy, breakthroughs in nanotechnology are expected to facilitate the development of other advanced applications in nanoelectronics, nanomedicine, nanomaterials (e.g., nanocomposites), nanoelectromechanical systems (NEMS), and nanorobotics.

A great deal of nanotechnology research in the U.S. comes under the purview of the National Nanotechnology Initiative (NNI) which provides a framework for government agencies to collaborate. Many government departments and agencies participate in the NNI including the Department of Energy (DOE), the Department of Defense (DOD), and the intelligence community including Homeland Defense. The NNI focuses on nine Grand Challenge areas; one of these Grand Challenge areas targets robotics.

It is of particular interest to the Intelligent Systems and Robotics Center (ISRC) to determine its role in the growing field of nanotechnology. Naturally and more specifically, the field of nanorobotics is the most pertinent topic of interest to the ISRC and is discussed in greater detail in this report.

1.2 Nanorobotics

Nanorobotics, sometimes referred to as molecular robotics, is an emerging research area as evidenced by recent topics in the literature. In general, nanorobotics carries a variety of definitions throughout the literature. Consequently, the field of nanorobotics can be generally divided into two main focus areas (Requicha, 1999; 2003).

The first area deals with the design, simulation, control, and coordination of robots with nanoscale dimensions (i.e., nanorobots). Nanorobots, nanomachines, and other nanosystems are objects with overall dimensions at or below the micrometer range and are made of assemblies of nanoscale components with individual dimensions ranging approximately between 1 to 100 nm. Much of the research conducted in this area remains highly theoretical at the present, primarily because of the difficulties in fabricating such devices. Although artificial nanorobots do not yet exist, nature's biological nanorobotic systems do exist and provide evidence that such systems are at least possible (Requicha, 2003). As a result, nanorobots have for the most part been explored in the biological context of nanomedicine.

The second area deals with the manipulation and/or assembly of nanoscale components with macroscale instruments or robots (i.e., nanomanipulators). A much greater number of research papers have been generated in this area. Due to the advances in nanotechnology and its rapidly growing number of potential applications, it is evident that practical technologies for the manipulation and assembly of nanoscale structures into functional nanodevices need to be developed. Nanomanipulation and nanoassembly may also play a crucial role in the development of artificial nanorobots themselves. Manipulation at the nanoscale is still in its infancy and the physical and chemical phenomena at this scale are not completely understood (Sitti, 2001a).

This report highlights the findings of an extensive review of the literature in the area of nanorobotics. The main goal of this survey is to identify accomplishments and advancements that have been made in this relatively new and emerging field.

1.3 Organization of the Report

The remainder of this report is organized as follows. Chapter 2 discusses the highly theoretical area of biologically inspired nanorobots. Chapter 3 highlights the most recent topics and advancements in robotic nanomanipulation. Chapter 4 describes the nanomanipulation system and hardware currently available at the ISRC. Finally, a summary of the survey and recommendations for future work are given in Chapter 5. All references surveyed in this report are listed in Chapter 6.

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2. Biologically Inspired Nanorobots

This chapter discusses topics in the field of biologically inspired nanorobots, also sometimes referred to as bionanorobotics. Nanorobots, nanomachines, and other nanosystems are objects with overall dimensions at or below the micrometer range and are made of assemblies of nanoscale components (Requicha, 2003). This field of nanorobotics studies the design, manufacturing, programming, and control of nanoscale robots. Nanorobots would constitute any active structure capable of actuation, sensing, signaling, information processing, intelligence, or swarm behavior at the nanoscale (Ummat et al., 2004b).

The envisioned nanorobotic applications range from medical to environmental sensing to space and military applications (Ummat et al., 2004a; 2004b). For example, nanorobots could be used to give precise drug delivery for repairing cells or to seek and destroy pathogens. Some researchers believe this would cause a paradigm shift from treatment to prevention in the medical community (Requicha, 2003).

Much of the research conducted in this area remains highly theoretical at the present, primarily because of the difficulties in fabricating such devices. Although artificial nanorobots do not yet exist, nature's biological nanorobotic systems do exist and provide evidence that such systems are at least possible (Requicha, 2003). As a result, nanorobots have for the most part been explored in the context of biology. Thus, a more active area of research in this field focuses on biomolecular machines which are inspired by "nature's way of doing things" at the nanoscale (Ummat et al., 2004a; 2004b). As our knowledge and understanding of biomolecular machines continues to increase, we move closer to the possibilities of using these existing machines as nanorobotic components or even fabricating artificial molecular machines from scratch as envisioned by Drexler (1992).

2.1 Biological Molecular Machines

The main goal in the field of biological molecular machines is to use various biological elements - whose function at the cellular level creates motion, force, or a signal - as machine components. These components could potentially be used to perform their preprogrammed biological function in response to specific physiochemical stimuli in an artificial setting. In this way, biological structures such as proteins could act as motors, mechanical joints, transmission elements, sensors, etc (Mavroidis and Dubey, 2003). It is possible that all of these different components could be assembled together in a proper manner to form nanodevices with multiple degrees of freedom able to apply force and manipulate objects in the nanoscale world (Ummat et al., 2004a).

Biomolecular motors have drawn a lot of attention because they operate at extremely high efficiencies (e.g., some near 100%), could be self-replicating, and are readily available in nature (Ummat et al., 2004a). The majority of natural molecular machines

are protein-based. Nature deploys proteins to perform various cellular tasks, from moving cargo to catalyzing reactions (Mavroidis et al., 2004).

The following sections summarize only a few examples of the motor proteins found in nature. Many other types of biological and artificial molecular motors exist and have been studied in greater detail. Refer to Ummat et al. (2004a; 2004b), Mavroidis et al. (2004), Kinbara and Aida (2005), and Balzani et al. (2000) for a comprehensive review of biologically-based molecular machines.

2.1.1 Myosin, Kinesin, and Dynein Motor Proteins

Motor proteins are tiny vehicles that transport molecular cargoes such as organelles, lipids, and proteins within cells. They also play a key role in driving the motion of muscles. These minute cellular machines exist in three families including the myosins, the kinesins, and the dyneins (Ummat et al., 2004a). Many protein-based molecular motors convert the chemical energy present in ATP into mechanical energy. Adenosine triphosphate (ATP) is a molecule that is the principal form of energy immediately usable by a living cell.

Myosin-based molecular motors transport cargo along “tracks” of actin filaments. Actin filaments are two-stranded helical polymers of the protein actin with diameters of about 5-9 nm. Myosin-based motors convert the energy available in ATP into mechanical energy by hydrolyzing it. This process of hydrolysis causes the protein to change shape (i.e., conformational change) thus pushing itself along the actin filament by about 10 nm per step. This is also used in the process of force generation during muscle contraction in which actin filaments and myosin filaments slide past each other (Ummat et al., 2004b).

Both kinesin and dynein are involved in cellular cargo transport along “tracks” called microtubules. Microtubules are tubes made of the protein tubulin with diameters of about 25 nm and are present in cells in an organized manner. Microtubules have different polarities associated with opposite ends. Kinesins move from the minus end to the plus end while dyneins move from the plus end to the minus end. Kinesin-based motors are unique because they are comprised of feet-like structures which, through ATP hydrolysis, give them the ability to literally “walk” along the microtubules. Kinesin is able to take about 100 steps before detaching from the microtubule while moving at 1000 nm/sec and exerting forces on the order of 5-6 pN. Dyneins are involved in both cargo transport as well as producing bending motions of cilia and flagella (Ummat et al., 2004b).

2.1.2 Flagella Motors

Certain single-cell bacteria such as *E. coli* are equipped with rotary motors that have diameters of about 45 nm. Each of these molecular rotary motors drives a helical-shaped filament that extends out of the cell and provides propulsion. In addition to this rotary mechanism, *E. coli* also have components that serve as particle counters, rate meters, and gearboxes. The flagella motors can propel bacteria as fast as 25 $\mu\text{m/s}$. A typical flagella motor from *E. coli* consists of about 20 different proteins. In most cases, the rotary motors are powered by a flow of protons through the cell membrane (i.e., proton-motive

force) which results from a build-up of a trans-membrane ion gradient. A complete explanation of how this proton flow is able to generate torque is not currently available. However, it is known that the proton-motive force is a result of the difference in pH between the inside and outside of the cell membrane. Thus, the speed of rotation of their motors depends on the pH of the surroundings (Ummat et al., 2004b).

2.2 Theoretical Design, Control, and Simulation

Since nanorobotic devices have not yet been fabricated, evaluating possible design and control algorithms requires the use of theoretical estimates and virtual environments (Ummat et al., 2004a). Sharma et al. (2005) discuss the use of molecular dynamics (MD) simulation and virtual reality (VR) in the field of bionanotechnology to help better understand the complex biological structures and mechanisms at the nanoscale. MD simulation is performed to help predict the dynamic performance of bio-components such as proteins (Ummat et al., 2004b). MD simulation can help gauge the feasibility of a particular conformation (shape) of a biomolecule through energy constraints. For example, a transition from one given state (i.e., shape) to another must be energetically favorable. Thus, MD simulation can aid in the evaluation of very complex force systems in which molecular conformational changes are directly related to the interactions of its individual atoms with each other and the environment.

Dubey et al. (2003) investigate the behavior of a novel biomolecular motor known as the viral protein linear (VPL) motor. The VPL motor is essentially a nanoscale actuator capable of producing motion of about 10 nm through conformational change. Dubey et al. (2004) and Sharma et al. (2004) further study the dynamics and kinematics of the VPL actuators using MD simulations. Furthermore, Sharma et al. (2004) present a methodology to determine the workspace of an amino acid chain (i.e., protein) through kinematic analysis. The use of a kinematic analysis can be used to suggest the possible geometric paths that could be followed during protein conformational transitions. Then, MD simulations can be carried out to narrow down the possibilities by pointing to the most energetically feasible paths. This method helps avoid the tedious and time consuming process of using MD simulations alone.

Even though artificial nanorobots do not yet exist, some work has been done in the study of coordination and control of large numbers (i.e., swarms) of such nanorobots. Cavalcanti (2003) introduces a novel simulation approach intended for the research of nanorobot control design. The simulator allows for a virtual 3D graphical environment in which nanorobots can be placed and can interact with other nanorobots or their environment. The simulated locomotion of the nanorobots was based on concepts from underwater robotics (i.e., nanorobots behave as submarines). Cavalcanti et al. (2004) expand on this simulation framework to investigate the design and control of medical nanorobots in a simulated blood vessel to treat stenosis (i.e., narrowing of the blood vessel). Cavalcanti and Freitas (2005) present control strategies utilizing genetic algorithms and neural networks for the control of the collective behavior of a group of nanorobots to carry out a specified task.

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3. Robotic Nanomanipulation

One of the biggest challenges in the field of nanotechnology is nanomanipulation and nanoassembly. Due to the advances in nanotechnology and its rapidly growing number of potential applications, it is imperative to develop practical technologies for the manipulation and assembly of nanoscale structures into functional nanodevices. Manipulation at the nanoscale is still in its infancy and the physical and chemical phenomena at this scale are not completely understood (Sitti, 2001a).

This chapter highlights the most recent topics and advancements found in the literature on robotic nanomanipulation. First, a brief overview is given of common imaging tools used in this research area including electron microscopy and scanning probe microscopy. The application and integration of these tools for robotic nanomanipulation are discussed from examples in the literature. Other various nanomanipulation and nanoassembly processes and tools found throughout the literature are discussed as well.

3.1 Electron Microscopy

3.1.1 Scanning Electron Microscope (SEM)

Since the commercial availability of the SEM in 1966, it has been a valuable resource for viewing samples at a much higher resolution and depth of field than the typical light microscope. Conventional SEMs can resolve down to the nanometer scale (~2 nm) whereas a light microscope can only resolve down to approximately 200 nm (Goldstein et al., 2003). Unlike conventional light microscopes, SEMs have a high depth of field which gives imaged samples a three dimensional appearance. Early SEMs were limited to viewing conductive samples. However, many of today's SEMs can image nonconductive samples in addition to conductive samples using variable pressure chambers.

Similar to the light microscope, the SEM contains an illumination source. This source, known as the electron gun, supplies the electrons that form an electron beam. As the electron beam proceeds down the electron column toward the specimen chamber, lenses (usually magnetic) are used to shape and channel the beam onto the surface of the specimen (Goldstein et al., 2003). The sample is typically mounted on a specimen stage which can be motion controlled with a joystick. The interior of the SEM contains a vacuum environment that supports beam formation and prevents the electrons from scattering.

After the beam has been shaped by lenses, a pair of deflection coils scan the beam over a rectangular portion on the specimen surface. The electrons strike the sample and cause it to discharge and/or scatter various levels of radiation. Detectors inside the chamber collect the emitted and/or scattered radiation levels. The detected signal will vary

depending on the magnitude of radiation received from the sample. This signal is then processed and used to form a real-time image of the specimen on a computer display.

3.1.2 Transmission Electron Microscope (TEM)

The TEM can resolve to an atomic scale of magnitude down to about 1 Å (i.e., 0.1 nm). The TEM mode of operation is similar to that of the SEM in that both microscopes contain an electron gun source of illumination. However, the TEM detects the electrons that pass through a given sample. As a result, the electron gun of the TEM operates at higher energy levels between 50-100 kV, while the SEM's electron gun operates around 1-20 kV. In order for proper imaging to take place, the sample must be very thin so that electrons from the beam can pass through the specimen. Electrons that do not pass through the sample cannot be detected. Different types of detectors can be used in TEMs. Two common types of detectors are fluorescent screens or photographic films. A denser portion of the specimen will allow fewer electrons to reach the detector, thus the denser portions of the specimen appear darker in the image. Unlike the SEM, the TEM produces images that are two dimensional in appearance (Ramberg and Siegel, 2001).

3.2 Scanning Probe Microscopy (SPM)

3.2.1 Scanning Tunneling Microscope (STM)

Similar to the TEM, the STM can also resolve specimens down to the atomic scale (Sellin, 2002; Wickramasinghe, 2001). The scanning probe of the STM is comprised of a noble metal sharpened to an atomic sized tip which is mounted to a piezoelectrically driven (x,y,z) linear stage (Müller et al., 1996; Schmid, 2005; Wickramasinghe, 2001). The STM makes use of the quantum mechanical effect known as tunneling. Electron tunneling occurs when electrons, driven by a small potential difference, flow across the gap between the probe tip and sample. This event takes place at Ångström-scale distances between the probe tip and the sample (Requicha, 1999). The tunneling current, which is typically on the order of a few nanoamperes, is directly related to the tip/sample separation distance. Thus, the tunneling current can be measured and is kept at a constant value by controlling the tip/sample gap distance (z) with a feedback control system. The probe tip is then scanned (x,y) along the entire surface of the sample. Since the control system maintains a constant tunneling current, and thus maintains a constant tip/sample distance (z), the result of a scan yields a z(x,y) terrain map of the sample with enough resolution to detect atomic-scale features (Requicha, 1999). The STM can achieve faster imaging by operating in so-called constant height mode in which the probe tip is scanned in a plane parallel to the average surface portion. The tip/sample distance (z) can then be inferred directly from the measured tunneling current (Binnig and Rohrer, 1999).

3.2.2 Atomic Force Microscope (AFM)

The AFM is considered to be a spin-off of the STM. One shortcoming of the STM is that it requires conductive probe tips and samples to work properly. The AFM was developed

in order to view nonconductive samples, giving it a wider applicability than the STM (Gerber, 2004; Requicha, 1999). In addition to imaging nonconductive samples, the AFM can also image samples immersed in liquid, which is useful for biological applications (Doktycz et al., 2003). Although the STM and AFM are similar in that they both scan the surface of a sample with an atomically sharp probe, they operate under slightly different principles. The AFM is based on interatomic forces as opposed to electron tunneling used in the STM. The AFM probe tip is mounted on the end of a microscale cantilever beam. At very close distances, the forces between atoms in the probe tip and atoms in the sample cause the cantilever to deflect. This deflection is usually measured by striking the back of the cantilever with a laser. The reflection of the laser beam hits a photodetector which can be used to decipher the deflection of the cantilever (Baselt, 1993). The force can then be calculated by using Hooke's law which simply relates the applied force to the stiffness and deflection of a material (Baselt, 1993; Wickramasinghe, 2001). The forces that are found are usually on the scale of pico-Newtons (10^{-12} N) (Binnig and Rohrer, 1999).

The AFM has three main modes of operation known as contact mode, non-contact mode, and tapping mode. In contact mode, the probe tip is scanned as it makes contact (i.e., within a few Å) with the surface of the sample (Requicha, 1999). Contact mode can operate either by using a constant force approach or a variable deflection approach. When operating in constant force mode, the probe tip is adjusted by the feedback control system to maintain a constant deflection (i.e., constant force), and thus maintains a constant height above the sample surface as it scans. The AFM records the height adjustments that are made to maintain a constant force and uses this information to create the sample image. The variable deflection mode permits the tip to scan over the sample without making any height adjustments to the cantilever. In this case, the measured deflection data of the cantilever is used to form the sample image (Nanoscience Instruments, 2002). Contact mode provides good resolution, however, it cannot be used with delicate samples (e.g., biomaterials) which can be damaged by the contact forces (Requicha, 1999). In addition, contact mode suffers from noise, adhesion, and tip wear problems (Requicha, 2003).

In non-contact mode, the cantilever oscillates very close (i.e., within several nm) to the sample surface without actually touching it. The tip is vibrated near the resonant frequency of the cantilever beam which is typically in the kHz range (Requicha, 1999). The attractive forces between the probe tip and the sample will cause changes in the resonant frequency and amplitude of the cantilever. These changes can be used to form the sample image (Nanoscience Instruments, 2002). Non-contact mode has poorer resolution than contact mode, but it can be used with delicate samples (Requicha, 1999).

Alternatively, the AFM utilizes another mode widely known as tapping mode (i.e., dynamic force mode or intermittent contact mode). This mode is a combination of contact and non-contact mode and exploits the advantages of both techniques. In tapping mode, the probe tip oscillates closer to the sample than in non-contact mode. The probe will also lightly and intermittently touch the sample. This type of imaging achieves very high resolution and can be used with delicate samples (Nanoscience Instruments, 2002). This is the preferred mode in many applications (Requicha, 2003).

Unlike the SEM and TEM, both the STM and AFM do not require a vacuum environment in order to function. However, a high vacuum is advantageous in order to keep the samples from becoming contaminated from the surrounding environment as well as controlling humidity (Schmid, 2005). In addition, atomic resolution in air is hardly possible with an AFM due to humidity. As a result of humidity, a water film is formed and creates problems because of capillary forces. This can be resolved by operating in vacuum environment or completely in a liquid solution (Binnig and Rohrer, 1999).

3.3 Nanomanipulation with the SPM

Although the SPM was originally intended for the imaging of samples, it was quickly recognized that it could be used to modify the samples as well. The SPM performs essentially like a 3-DOF robot. Conventional SPMs can translate in (x,y,z) space, but cannot orient their probe tips. There are various problems related to the operation of the SPM. These problems are mainly due to sources of spatial uncertainty including tip geometry effects as well as drift, creep, and hysteresis issues with the piezoelectric actuators (Requicha et al., 2001b; Requicha, 2003).

Since SPM probes do not have infinitesimally small tips, a geometrical convolution of the probe tip and the sample occurs as the tip scans across the sample surface. This has an effect similar to low-pass filtering which effectively appears as a broadening of sample features in the resulting image. This has led to a significant amount of work investigating the use of carbon nanotubes as SPM probe tips because of their high aspect ratios and extremely small diameters (Milas et al., 2002). In addition to this, the shape of the probe tip may not be precisely known and can vary due to wear during operation.

Thermal drift between the probe tip and the sample is a major cause of spatial uncertainty in the SPM. Room temperature drift of one atomic diameter per second is common (Requicha, 1999). Thus, for atomic-scale operations, it is not unlike picking up parts from a moving conveyer belt. Thermal drift is negligible at very low temperatures, and all experiments in atomic-precision manipulation to date have been done at extremely low temperatures (~ 4 K). The issue of thermal drift is addressed by Mokaberi and Requicha (2004) which highlights efforts towards automatic drift compensation through the use of Kalman filtering techniques.

Creep, hysteresis, and other nonlinearities in the piezoelectric actuators also contribute to spatial uncertainty in the SPM (Sitti, 2004; Requicha, 1999; 2003). This becomes a difficult problem to correct since the x- and y-motion over small regions is controlled primarily in open-loop because of a lack of suitable sensors than can be used in a feedback scheme (Requicha, 1999). The z-motion, however, is very accurate because of the tunneling current (e.g., STM) or force (e.g., AFM) feedback.

A large portion of the existing literature on robotic nanomanipulation discusses the use of piezoelectrically driven probes to manipulate nanoscale objects. In this type of research, researchers generally use systems that incorporate one or a combination of the following microscopy technologies: scanning electron microscopy (SEM), transmission electron microscopy (TEM), scanning tunneling microscopy (STM), and atomic force microscopy

(AFM). Although the majority of nanomanipulation processes found throughout the literature are carried out through the use of an AFM, a significant portion of work has also investigated the use of custom-designed and/or commercially available nanomanipulators. The following sections summarize research work investigating various techniques and tools for the nanomanipulation of nanoscale objects.

3.4 Manipulation of Atoms

The first real example of nanomanipulation was demonstrated by Eigler and Schweizer (1990) who manipulated individual xenon atoms using a STM to form the name of their employer, “IBM”. Atomic manipulation is typically performed in an ultra-high vacuum (UHV) environment and at very low temperatures (~ 4 K) to prevent contamination and drift issues (Requicha, 2003). In addition, atomic manipulation only works for certain combinations of surfaces and atoms (Eigler, 1999). Because of this, the idea of building three dimensional nanoscale structures atom-by-atom under these limiting types of conditions does not appear to be very practical at the moment.

3.5 Manipulation of Nanoparticles

An alternative and more typical approach to the assembly of nanoscale structures has been to work with relatively larger sized building blocks in ambient conditions. As such, much work has investigated the manipulation of various nanoparticles with AFM probes. The manipulation of nanoparticles may play an important role in the assembly and building of prototype nanoelectronic devices (Requicha, 2003) as well as devices for photonics applications (Garcia-Santamaria et al., 2002).

There are several protocols for particle manipulation by pushing with an AFM probe, all of which share the following aspects (Requicha, 2003). First, an image of the sample is taken to determine where the particle is located on a substrate. Then, the probe tip is moved against the particle and the AFM operating parameters are changed so that a force higher than that used for imaging is applied. Typically, for particle pushing, the AFM is operated in dynamic force mode with the force feedback turned off. The AFM probe is moved in a straight line that goes through the center of the particle. Requicha (2003) claims that this protocol is almost 100% successful when the AFM probe tip is sharp (radii on the order of 10-20 nm) and the tip pushes the particle very close to the center. In this work, successful pushing has been accomplished in ambient air or in liquids at room temperature without strict environmental controls. For more details regarding the work done at the Molecular Robotics Laboratory at the University of Southern California using the AFM to push particles, refer to Requicha et al. (1998; 2001b), Requicha (1999; 2003), and references therein.

Decossas et al. (2003) investigate the manipulation of spherical silicon nanoparticles with diameters varying from 4-30 nm with an AFM tip. In this work, three nanomanipulation techniques are presented. The first technique uses electrostatic attraction to pick up a particle and deposit the particle at a different location on a substrate (i.e., what the authors refer to as “nanocrane”). A particle can be picked up by the repeated application

of negative and positive tip voltages. It is assumed that this process charges the particle and therefore causes electrostatic attraction between the particle and probe tip. Then the particle can be moved to a different location and deposited by repeating the same process (i.e., applied positive and negative voltages). The second method investigates the simultaneous manipulation of a line of nanoparticles by rapidly sweeping the AFM tip perpendicular to the direction of slower forward motion. Using this method, it was shown that a sparse “field” of particles could be manipulated into dense lines (i.e., referred to as “nanobroom”). The last technique investigates the mechanical pushing of individual nanoparticles to form dense lines as well as a circle made of six nanoparticles down to a precision of about 10 nm.

A fair amount of work has been done in the modeling of nanoscale interaction forces and dynamics during pushing operations of particles with AFM probes (Hashimoto and Sitti, 1999; Sitti and Hashimoto, 2000a; 2000b; Sitti, 2001b; Tafazzoli and Sitti, 2004). Tafazzoli and Sitti (2004) investigate the dynamic behavior of nanoparticle motion during probe-based manipulation. The authors discuss various dynamic situations in which a spherical nanoparticle will stick, slide, or roll when pushed with an AFM probe tip. Sitti (2001b) gives preliminary experimental results showing that these different frictional behaviors can be observed with a custom-made AFM system. Sitti and Hashimoto (2000a) demonstrate an AFM nanomanipulation system which can successfully position submicron latex particles with a precision of about 30 nm.

Most particle nanomanipulation work has involved the manipulation of nanoparticles on planar two dimensional surfaces. Requicha et al. (2001a) investigates the layered nanoassembly of three dimensional structures using a combination of planar AFM nanomanipulation and sacrificial surface layers. The basic method behind this technique is to construct a three dimensional nanostructure by building up two dimensional slices of the structure layer by layer. First, nanoparticles are placed at desired locations on a flat substrate. Then, a sacrificial layer with a height equal to that of the nanoparticles is deposited over the substrate. The sacrificial layer must adhere to the substrate but not to the nanoparticles. Additional nanoparticles are placed upon the new sacrificial layer and manipulated to desired locations with an AFM. The sacrificial layer must then be removed without damaging or disturbing the nanoparticles already placed. In this work, a 5 nm particle is successfully assembled directly on top of a previously deposited nanoparticle using the technique described above. Experimental results of this work indicate that layered assembly is possible; however additional work is required on the development and optimization of materials and deposition techniques.

Another example of layered assembly is given by Garcia-Santamaria et al. (2002) in which a custom nanomanipulator inside an SEM (Morishita and Hatamura, 1993) is used to assemble diamond-type lattice structures from 0.9 μm diameter silica spheres. These lattice structures have potential applications in the areas of photonics and biological tissue engineering. In this work, lattices are constructed layer-by-layer by placing individual silica spheres in designated locations (e.g., FCC or BCC configurations) and inserting latex spheres as a temporary supporting scaffold. The first layer is constructed upon a patterned substrate to ensure appropriate spacing between spheres. Subsequent layers are formed by placing individual spheres at stable locations on top of supporting

spheres from the previous layer. The sacrificial latex spheres are then removed without disturbing the silica spheres by an oxygen plasma etching process. The authors demonstrate the assembly of a six-layer structure made of 133 latex spheres and 274 silica spheres. In addition, this work also shows that silica spheres can be “glued” together by focusing an electron beam on their junction for a few seconds (i.e., electron-beam-deposition).

3.6 Manipulation of Carbon Nanotubes

The development of new and improved fabrication techniques has allowed the bulk manufacture (i.e., large-scale synthesis) of various types of nanoscale carbon structures known as fullerenes (Terrones et al., 1996; Terrones et al., 2001). Fullerenes, which are molecules made from cage-like structures of carbon atoms, can take on various sizes and geometries including spherical, toroidal, polyhedral, star-like, helical, and tubular shapes.

One nanostructure of significant interest is the carbon nanotube (CNT) which began gaining widespread interest in the early 1990s following the work of Iijima (1991). Since then, the number of yearly nanotube publications has increased exponentially to over 1500 publications in 2001 alone (Baughman et al., 2002; Terrones, 2003). Carbon nanotubes have been proposed for use in numerous applications due to their remarkable electrical, mechanical, and thermal properties (Baughman et al., 2002; Terrones, 2003; Terrones et al., 1999; Terrones and Terrones, 2003; Ajayan and Vajtai, 2001). These include applications in high-strength composite materials, scanning probe microscopy, field emission sources, nanoelectronics, nanoelectromechanical systems (NEMS), nanorobotics, chemical sensors, bio-nanotechnology, and energy storage.

Because of their unique properties, CNTs have been proposed as prototypical nanoscale building blocks (Madsen et al., 2003). A significant amount of research has investigated the manipulation of CNTs as evidenced by the large number of publications in this area. The atomic force microscope (AFM) has been one of the most common tools used for the manipulation of CNTs (Wong et al., 1997; Hertel et al., 1998; Postma et al., 2000b; Williams et al., 2002; Zhang et al., 2002; Shen et al., 2002; Liu et al., 2003). Manipulation with AFM probes typically involves contact pushing in order to perform actions such as sliding, rolling, bending, kinking, and cutting of CNTs. These types of manipulation processes have been used mainly for the characterization of the mechanical and electrical properties of CNTs (Wong et al., 1997; Postma et al., 2000a; Tomblor et al., 2000; Williams et al., 2002), the study of interactions between CNTs, substrates, and other CNTs (Hertel et al., 1998; Postma et al., 2000b; Zhang et al., 2002; Shen et al., 2002; Liu et al., 2003), and the construction of prototype nanoelectronic devices (Roschier et al., 2002; Thelander and Samuelson, 2002).

Generally, AFM technology is mostly limited to planar (2D) manipulations on a substrate (Fukuda et al., 2003; Dong et al., 2001a). Thus, work has focused on the development of more complex 3D nanomanipulation systems (up to 16 DOF) which consist of one or more probes that have been integrated into scanning electron microscopes (SEM) for real-time visual feedback (Yu et al., 1999; 2003; Guthold et al., 2000; Dong et al., 2001; 2002; 2003; 2004; Fukuda et al., 2003; 2004; Skidmore et al., 2004; Arai et al., 2004).

These systems have proven to be useful for the 3D manipulation, testing, and characterization of CNTs.

Fukuda, Arai, and Dong of Nagoya University in Japan are responsible for a significant portion of publications in the field of robotic nanomanipulation. The authors have established a prototype nanomanipulation system referred to as “Nanolaboratory”. This custom-built system is seen throughout various related publications on the manipulation and testing of CNTs for several different applications. The system consists of a custom-designed nanomanipulator which is mounted inside a field emission scanning electron microscope (FESEM) for real-time visual feedback (Fukuda et al., 2004a; 2004b; 2004c). The nanomanipulation system is comprised of four individual units which give the system a total of 16 DOF. The first unit is a 3-DOF sample stage that allows lateral and angular motion. The remaining three units can be equipped with 3-4 AFM cantilevers which are used as end-effectors, each of which allow for coarse and fine positioning. This nanomanipulation system has been used for the construction and testing of various CNT devices including nanotube scissors (Dong et al., 2002b), CNT mass flow sensors (Fukuda et al., 2004b), CNT-based position sensors (Liu et al., 2004), and CNT linear bearings (Fukuda et al., 2004a). It has also been used for the testing of nanoscale force measurements (Arai et al., 2002; 2003), CNT shape modification methods (Dong et al., 2002b; 2003a; 2004), and CNT field emission characterization (Arai et al., 2004).

Nakajima et al. (2004a; 2004b) develop a hybrid robotic nanomanipulation system in which they incorporate the use of both a TEM and SEM for imaging. The TEM does not contain enough space to house the positioners required to perform the desired manipulations. In addition, the SEM does not have the resolution needed for the authors’ purposes. In order to fully utilize both microscopes’ capabilities, the authors combine the TEM and SEM in their nanomanipulation system in order to take advantage of the SEM’s large specimen chamber and the TEM’s high resolution capabilities. The TEM manipulator has 3 DOF while the SEM manipulator has a total of 8 DOF. In order to perform manipulations, the TEM manipulator is first incorporated with the SEM manipulator inside the SEM chamber. The SEM manipulator is used to place the samples on the TEM manipulator stage and to adjust the distance between the samples. After samples are properly placed, the TEM manipulator is installed inside of the TEM chamber to perform fine positioning and high resolution imaging. This system has been used to experimentally study the telescoping behavior of multi-walled CNTs.

3.7 Manipulation of Biological Materials

The use of SPMs to image and manipulate biological samples has had a tremendous impact on the field of biology in recent years. Studies of single molecules by SPMs are advantageous because they can provide intrinsic properties of the individual molecules themselves as opposed to just the bulk properties of larger samples. SPM technology has become an invaluable tool for the understanding of biological structures and processes at the nanoscale. Examples of AFM applications in imaging and nanomanipulation include the extraction of chromosomal DNA for genetic analysis, the disruption of antibody-

antigen bonds, the dissection of biological membranes, and the nano-dissection of protein complexes (Fotiadis et al., 2002).

Hu et al. (2004) and Lu (2004) investigate the use of an AFM to manipulate single DNA molecules on a substrate. For both works, the DNA samples were initially prepared with a process known as “molecular combing” to remove any random coiling of the DNA. After the molecular combing process, the DNA strands are arranged linearly on the substrate. Then the AFM probe tip can be used to image (i.e., under tapping mode) and manipulate the DNA strands. Lu (2004) investigates the use of the AFM tip to kink, cut, and displace parts of a DNA strand. Hu et al. (2004) demonstrates the ability to spell out the letters “DNA” with the DNA strands through nanomanipulation. In addition to this, folding of the DNA strands was stimulated by pushing near the ends of the DNA with the AFM tip. Potential applications for this type of technology in gene research include the direct detection of DNA mutation and sequencing of selected pieces of DNA. Refer to Fotiadis et al. (2002) and references therein for a comprehensive review of imaging and manipulation of biological structures with the AFM.

Imura et al. (2002) develops a nano-surgery system for cell organelles such as chloroplast and mitochondria. The typical size of chloroplast is approximately 1 μm while the size of mitochondria is between 0.5-1 μm . The system uses a glass pipette with an inner diameter of 420 nm and outer diameter of 720 nm to capture or discharge a target organelle. The system consists of a sample stage, microscope, and nano-pipette which is attached to a 3-DOF actuator. Experiments show the successful capture of mitochondria from a human-lung-cancer cell.

Mathews et al. (2004) describe the design and fabrication of a temperature-controlled electromagnetic needle (EMN) to generate custom magnetic field gradients for biomedical applications. The EMN is capable of applying large static or dynamic forces (1-50 nN) to magnetic nanoparticles. Needle tips with radii between 100 nm and 20 μm were investigated. It was found that larger tip radii can be used to apply large forces to multiple particles over a large region. Alternatively, smaller tip radii can be used to confine the magnetic force to within a few microns of the needle tip to selectively capture a single particle from a large population of similar particles. Experiments show that the EMN can selectively capture magnetic particles with diameters of 250 nm. Thus, this method could be used to manipulate, probe, and position magnetic particles linked to biological molecules or living cells.

Layton et al. (2004) present a methodology for the simultaneous mechanical testing and AFM imaging of single collagen fibrils under load. Collagen is the most abundant protein in the body and is common to all biological tissues. Collagen fibers, which have diameters in the range of 20-500 nm, demonstrate enormous physical strength and thus have been proposed as a biocompatible material for making nano-sutures. In this work, a Zyvex S100 nanomanipulation system was used in combination with an AFM for the mechanical testing of single collagen fibrils. One fiber end was glued to the Zyvex nano-probe while the other end was gripped under a light microscope. Tensile tests were then performed to determine the strength of the collagen fibrils. In addition to this, two techniques were proposed for tying knots in single fibrils as a step towards nano-suturing.

3.8 Nanoscale Gripping

3.8.1 Optical Tweezers

It is known from physics and the early history of optics that light has linear and angular momentum, and therefore could exert radiation pressure and torques on physical objects (Ashkin, 2000). Optical trapping was first discovered by Ashkin (1970) who showed that the forces due to radiation pressure from focused laser beams could be used to accelerate, decelerate, and even stably trap small micron-sized neutral particles. Through this work, two basic light pressure forces were identified. These two forces are known as the scattering force in the direction of the incident beam and the gradient force in the direction of the intensity gradient of the beam. Over the years, these laser trapping and manipulation techniques have been investigated and applied over a wide range of particle types including atoms, molecules, submicron particles, and macroscopic dielectric particles hundreds of microns in size. The use of optical manipulation techniques has made revolutionary contributions in the fields of physics, chemistry, and biology. Refer to Ashkin (2000) and references therein for a detailed explanation of the phenomenon as well as a comprehensive review of the history of optical trapping and manipulation of particles.

Chaumet et al. (2002) presents a detailed theoretical study of selective manipulation of nanoscale particles in air above a substrate using optical forces. In this study, a substrate is illuminated by two laser beams which create two counter-propagating evanescent waves. A tungsten probe is used to scatter these two waves and thereby generates a localized optical trap. A nanoscale object can be selectively brought into the trap and manipulated with the probe. This work discusses the influence of the geometry of the particles and the probe on the efficiency of the trap.

3.8.2 Nanotweezers

To accomplish pick-and-place manipulation and assembly, a reliable method of gripping is required. Gripping at the nanoscale is a challenge because it is difficult to control the balance of forces between the object, the surface, and the tool (gripper) where van der Waals and electrostatic forces may dominate (Molhave et al., 2004a). It has been shown that the interaction between an AFM probe tip and a CNT is strong enough to successfully transfer a vertically aligned nanotube from a substrate onto an AFM tip with no externally applied gripping forces (Hafner et al., 2001). However, a reliable gripping method should be applicable to CNTs in any arbitrary position or orientation on a surface. A few groups have demonstrated the fabrication and use of nanotweezers to successfully manipulate various nanostructures (Molhave et al., 2004a; Nakayama and Akita, 2001; 2003; Akita and Nakayama, 2002; Nakayama, 2002; Kim and Lieber, 1999). Unfortunately, nanotweezers can have problems releasing the nanostructures when opened.

Kometani et al. (2005) successfully fabricate three dimensional grippers with two, three, and four fingers using focused ion beam chemical vapor deposition (FIB-CVD) on the

end of a glass micropipette. The general sizes of the grippers are on the order of a couple microns. The grippers are electrostatically actuated by applying large voltages between 300-1200 V. The gap distance between fingers opens from about 0.1 to 2 μm with an applied voltage of 300 V. A latex sphere with a diameter of 1 μm was successfully captured by the four finger gripper. However, the authors make no mention of a successful release. In addition, they fail to comment on the field effect of the large bias voltage used to actuate the gripper on the latex sphere.

Wang et al. (2004) describe the fabrication and use of a thermally actuated gripper inside an SEM. The gripper consists of three independently actuated fingers. Each finger is actuated with a thermal bimetallic strip. An applied current causes resistive heating which, in turn, causes the fingers to move in the vertical direction. The finger tips are fabricated using conventional microlithography techniques as well as focused ion beam (FIB) milling to achieve below 100 nm spacing between each finger. Results from testing indicate that thermal actuator is able to generate a vertical displacement of 1 μm with an actuation power of only 0.14 mW. Thermally induced displacements as large as 20 μm have been achieved with an actuation power of 3.8 mW. In this work, it is shown that the thermally actuated grippers can be used to selectively grab a 500 nm diameter and a 40 nm diameter multi-walled CNT from an unorganized cluster.

Maruo et al. (2004) investigate optically-driven nanotweezers with two degrees of freedom. The nanotweezers are fabricated using a microstereolithography process and have probe tips measuring 1.8 μm in length and 250 nm in diameter. The tweezers consist of two independent lever arms which are pinned to the substrate on one end. The tweezers are driven by using a laser scanning manipulation technique. A small protruding part of the moveable arm called the “optical trapping point” is trapped by a focused laser beam and is maneuvered by scanning the laser beam to induce the desired type of motion. By scanning the laser beam in arcs, the target arm of the tweezers is subjected to an attractive force along the path of the laser beam, thereby opening and closing the tweezers. In addition, the applied torque on the tweezer arms were adjusted in the range of fN to pN by changing the intensity of the laser beam. Optimization of the shape of the optical trapping point was also investigated in this work.

Kim and Lieber (1999) develop functional nanotweezers with two arms made from carbon nanotubes. The tweezers are fabricated by attaching carbon nanotubes to independent electrodes on the tip of a tapered glass micropipette with an end diameter of 100 nm. Once fabricated, the arms of the tweezers are about 4 μm long, 50 nm in diameter, and spaced about 1 μm apart from each other. The tweezers are electrostatically actuated by applying a bias voltage to the electrodes. The tweezers can be fully closed by applying a bias voltage of 8.5 V. However, due to van der Waals interaction between the tubes, the tweezer arms remain closed after removal of the actuating voltage. They can be opened again by applying a voltage of the same polarity to both tweezer arms. Experiments demonstrate the successful capture and manipulation of a 500 nm cluster of polystyrene beads. In addition, the ability to grasp and remove an individual GaAs nanowire from an entangled bulk sample was also demonstrated.

Akita and Nakayama (2002) and Nakayama (2002) also present work on the development of nanotweezers made with carbon nanotube arms. The main difference between the approach here and the approach given by Kim and Lieber (1999) is that the carbon nanotubes arms are attached to silicon probes for the easier integration into an AFM. Experiments show that the AFM / nanotube tweezer system can successfully manipulate a 200 nm diameter silica particle as well as a carbon nanotube with a diameter of 20 nm and length of 1 μm on a silicon substrate.

Boggild et al. (2001) present a novel electrostatically actuated nanotweezer device where the shape of the tweezer tips can be customized depending on the application. The electrostatic actuators are fabricated using silicon microfabrication techniques. The unique design of this device avoids the application of an electrical voltage directly between the tweezer tips, thereby reducing electric field effects which could influence the gripping of a nanoscale object. The nanotweezer tips are fabricated using electron beam induced deposition which leads to the formation of 40-100 nm diameter carbon-based tips. This process can form converging tips with gaps down to 20 nm. The max actuation range for this device was found to be approximately 335 nm with the application of a 30 V bias voltage. Experimental support evaluating the gripping performance for this nanotweezer device is given by Molhave et al. (2004a). It was demonstrated that the tweezers are capable of picking up silicon nanowires from a substrate. However, the tweezers were unable to successfully grasp and pull a carbon nanotube from an entangled cluster.

3.8.3 Dielectrophoresis

Dielectrophoresis may be an alternative and viable means of gripping nanoscale structures. Dielectrophoresis (DEP), the use of non-uniform electric fields to manipulate polarizable objects, has been commonly used in the field of biology for the manipulation of micron sized objects such as biological cells (Burke, 2004; Hughes, 2000). The use of DEP at the nanoscale has exciting potential, however, it is still considered to be in its infancy (Burke, 2004).

Several groups have used DEP to successfully manipulate and orient CNTs in liquid solution between electrodes (Burke, 2004; Nagahara et al., 2002; Dimaki and Boggild, 2004; Tang et al. 2003; 2004; Zhang et al., 2004; Han et al., 2004). To the authors' best knowledge, only one other group (Fukuda et al., 2003; Dong et al., 2001a) has studied the nanomanipulation of individual CNTs by dielectrophoresis using a charged probe in a vacuum (not in a liquid suspension). In their work, an analysis is given comparing the dielectrophoretic gripping force and the van der Waals adhesive force of the CNT to the surface.

3.9 Joining Nanostructures

3.9.1 Nanosoldering

Electron beam induced deposition (EBID), also sometimes referred to as additive lithography (Koops et al., 1994) or nanowelding (Yu et al., 1999), is a method by which an electron beam can lay down carbonaceous material by disassociating organic molecules present in an electron microscope. Originally observed in the form of SEM-induced specimen contamination, it has since been turned from a “bug” into a “feature”. EBID has been used to produce various three dimensional nanoscale structures with features down to 5 nm (Koops et al., 1994; Molhave et al., 2004b) including nanotweezer tips with diameters as small as 40 nm (Boggild et al., 2001). Different types of compounds can be used in the EBID process resulting in a variety of deposited materials including tungsten, gold compounds, copper oxide, and platinum compounds. A comprehensive review of additive lithography using EBID is presented in Koops et al. (1994).

Although EBID has been used as a lithographical fabrication process for the building up of nanoscale structures, it has also been extremely useful for soldering nanostructures together. EBID has been used to join CNTs to probe tips for SPM applications (Milas et al., 2002), join CNTs to EBID structures and other CNTs (Molhave et al., 2004a), and attach CNTs between microelectrodes to test electrical and mechanical properties (Yu et al., 1999; 2000; Madsen et al., 2003; Molhave et al., 2004b). The soldering bonds were found to be mechanically stronger compared to the CNTs themselves.

Dong et al. (2001b; 2002a) introduce the concept of parallel EBID using CNTs as the electron beam emitters. An array of nanotube emitters could be fabricated and used to generate several electron beams simultaneously. The authors experimentally verify the proof of concept as they show that a single multi-walled CNT beam emitter can be used to successfully deposit material on a substrate. A more detailed study on the field emission characterization of CNTs is given by Arai et al. (2004).

3.9.2 Nanowelding

The process of welding unites materials together by causing them to flow together. In what may be true nanowelding, Terrones et al. (2000; 2002a; 2002b; 2002c) describe a mechanism by which single-walled CNTs are welded together under an electron beam at elevated (i.e., annealing) temperatures. In this case, there is no deposition of additional material. Instead, the electrons knock atoms loose, promoting rearrangement and “coalescence” of the tubes into welded molecular junctions. Molecular dynamics simulations were carried out in order to understand the formation mechanism of the molecular junctions. The formation of X-, Y-, and T-shaped junctions were theoretically studied and experimentally investigated.

Cumings and Zettl (2000) study the application of multi-walled CNTs as low friction linear bearings. In this work, “spot-welding” was used to attach the inner core of a multi-

walled CNT to a probe by means of a short and controlled electrical current pulse. Little is known about the exact nature of the spot-weld junction, but the authors presume it is a result of carbon-carbon bonds between the CNT and the tool tip. Obviously, this welding method requires electrical contact between two conductive structures.

A similar approach is used by Stevens et al. (2000) and Nguyen et al. (2001) to attach multi-walled CNTs to AFM probe tips. A high electric current is applied across the tip electrode which acts as a resistor to locally generate high temperatures. It is thought that this high temperature enables the formation of carbide at the contact site as well as causes the CNT to partially disassociate at the interface to deposit amorphous carbon.

3.9.3 Gluing

Hafner et al. (2001) study the high-yield assembly of CNT tips for SPM applications. In this work, CNTs are attached to AFM tips through the use of a UV-curable adhesive (Loctite 3105). This gluing process has allowed for reliable AFM imaging in fluid solutions.

3.9.4 Sintering

The use of a sintering process to connect latex nanoparticles is mentioned in Requicha (2003). In this process, the particles are first manipulated to desired locations near to each other on a substrate. Then, the particles are heated so that they melt together to form a single nanostructure. In this work, 12 latex particles with diameters of about 100 nm are manipulated and sintered to form the shape of a disk.

3.9.5 Chemical Bonding

Requicha (2003) also describes work investigating the use of chemical bonding to connect gold nanoparticles through the use of di-thiols. Di-thiols are organic molecules with sulfur end groups. The di-thiols self-assemble to the gold and serve as a chemical glue. The author demonstrates two variants to this approach. In the first approach, the particles are positioned and then immersed in the di-thiol solution to link them. In the second approach, the di-thiols are applied first, and then the particles are manipulated into contact, thus linking them. In this work, it was also shown that it is possible to push a group of linked nanoparticles as a whole.

Dong et al. (2002a; 2003) discuss experimental work in which multi-walled CNTs are joined together end-to-end. Through model analysis and experiments, the authors verify that the connecting force is most likely due to chemical (i.e., covalent) bonds between the carbon atoms and not due to weaker forces such as van der Waals.

3.10 Cutting Nanostructures

3.10.1 Mechanical Cutting

Zhang et al. (2002) investigate the manipulation and cutting of CNTs with an AFM probe. The authors are able to successfully separate a group of entangled CNTs as well as cut an individual CNT in half. The ability to cut the CNT greatly depends on the way the CNT is affixed to the substrate. In order for cutting to take place, the adhesion force effectively “pinning” the CNT to the substrate must be greater than the strength of the CNT itself. Otherwise, the AFM probe will only be able to bend, kink, or displace the CNT on the substrate. Hertel et al. (1998) also demonstrates the ability to cut CNTs in this fashion.

Dong et al. (2003a) investigates the length control of CNTs through mechanical strain. The two ends of a multi-walled CNT are attached to a fixed substrate and a movable probe through EBID. The probe is pulled away from the substrate in order to stress the CNT. To obtain a desired length, the location where the CNT breaks must be controlled. It is well known that CNTs will deform (i.e., flatten) on a flat substrate due to van der Waals forces. This technique takes advantage of these deformations which cause stress concentrations near the deformed regions on the substrate. It is most likely that the CNTs will break at these stress concentrations. Thus, the desired lengths can be controlled by changing the amount of CNT overlap on the substrate before it is attached and pulled.

Dong et al. (2002b) describe the design and fabrication of nanotube scissors. The nanotube scissors are created by attaching two multi-walled CNTs to a gold-coated AFM cantilever by EBID. The shape of each CNT is modified through the use of a custom-built robotic nanomanipulation system and EBID to form pre-stressed kinks and bends in the CNT. The opening and closing motion of the scissors is controlled by applying a current to the attached nanotubes. The nanotube scissors could potentially be used to cut other CNTs or nanowires.

3.10.2 Electron Ablation

Nakayama and Akita (2003) use method known as electron ablation to adjust the length of a CNT once it is attached to an AFM probe. In this work, electrons are emitted from another nearby CNT by field emission. A 0.5-2 μm gap is maintained between the two CNT tips throughout the process. A voltage of about 200 V is applied across the two CNT electrodes. After this procedure is repeated a few times, it can be observed that the CNT shortens in length. Although the mechanism for this process is not completely understood, it is possible that the phenomenon may be due to the excess electrons at the bonding sites, the lattice vibration due to Joule heating, and the field effect of the positive electrode which may cause instability in the C-C bonds at the end of the CNT. According to the authors, the most probable mechanism for electron ablation is a combination of the electronically and thermally induced bond instabilities as well as the field effects. A similar approach is used by Dong et al. (2003a) to modify the lengths of multi-walled CNTs.

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4. The Zyvex Nanomanipulation System

Currently, the ISRC has its own nanomanipulation system on-hand which is readily available to further investigate nanomanipulation and/or nanoassembly at Sandia. This chapter describes the nanomanipulation system in greater detail as well as discusses the results of preliminary testing of the system.

4.1 The Zyvex S100 Nanomanipulator

The Zyvex S100 is a commercially available nanomanipulator produced by Zyvex Corporation. The Zyvex S100 is designed to grasp, move, test, and position microscale and nanoscale samples. Our system consists of the Zyvex S100 and a LEO 1430 VP (variable pressure) scanning electron microscope which is shown in Figure 4.1. Although the LEO 1430 VP is a variable pressure SEM, the variable pressure detector was removed because it mechanically interfered with the operation of the S100.

One of the benefits of the S100 system is that it can be easily installed into an SEM or optical microscope which allows the operator to simultaneously view and manipulate samples. This setup is advantageous over general STM or AFM systems which can only perform imaging or manipulation, but not both at the same time. In addition to its manipulation capabilities, the S100 is capable of performing mechanical and electrical characterization by integrating sensors or probes into the connectors provided.

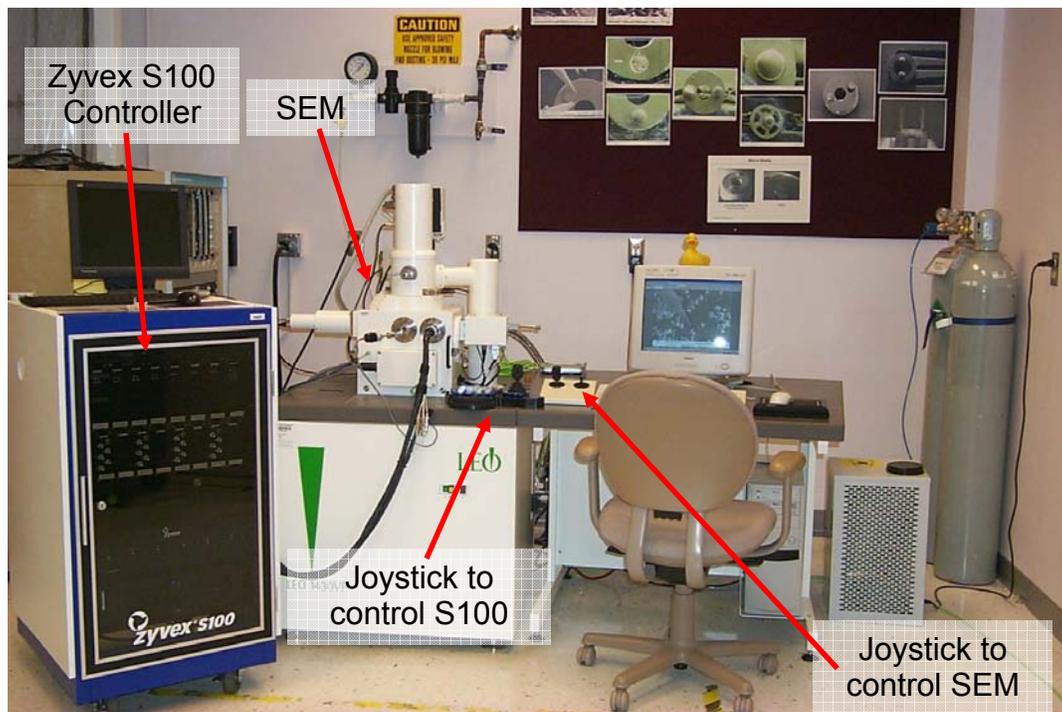


Figure 4.1. The Zyvex Nanomanipulation System

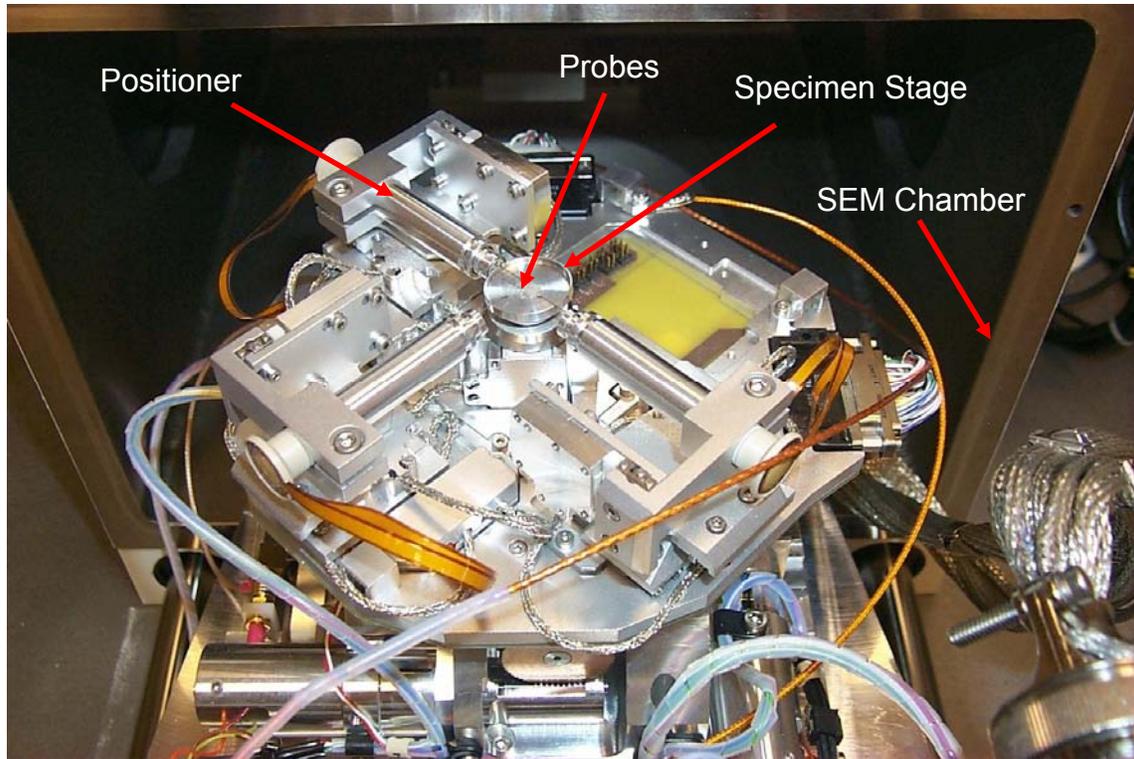


Figure 4.2. Zyvex S100 Mounted on SEM Stage

Figure 4.2 shows the Zyvex S100 nanomanipulator which is directly mounted on the SEM stage. The nanomanipulator typically consists of four positioners. However, one of the positioners was removed because it interfered with the backscatter electron detector. Each 3-DOF positioner allows for course and fine (x,y,z) motion. The course motion actuators permit a range of motion of 12 mm while the fine motion piezoelectric actuators provide 2 nm resolution fine motion. In addition, the S100 has a center rotational specimen stage to orient samples.

Figure 4.2 also shows the end-effector probes attached to the three positioners. A probe is attached by inserting the end into one of five connectors on the face of each positioner. Each of these five connectors can be used to independently drive and record electrical signals through the Zyvex controller cabinet. Probes were both purchased from Zyvex Corporation and fabricated at Sandia. The probes produced at Sandia were fabricated from tungsten wire using an electrochemical etching process capable of producing atomically sharp probe tips. The Zyvex probes are composed of polycrystalline tungsten wire with a 0.25 mm shank diameter and 50 nm tip radius.



Figure 4.3. Zyvox S100 Controller Cabinet

The controller cabinet for the Zyvox S100 is shown in Figure 4.3. The control system consists of a PC which provides signals to the driving amplifiers for each motion stage. The PC runs a Windows® environment which allows the operator to control each stage using a joystick and also allows the user to adjust a myriad of system operating parameters.

Also located inside the controller cabinet is the patch panel rack shown in Figure 4.4. The patch panel can be used to apply or detect analog signals from each of the probes inserted into the connectors on each of the positioners.



Figure 4.4. Zyvox Patch Panel Rack

4.2 Preliminary Testing

Preliminary testing of the Zyvox S100 nanomanipulator involved the manipulation of nickel particles with diameters of approximately 60 μm . Figure 4.5 shows a SEM image of a nickel particle lifted by the Zyvox fabricated probe. The image reveals that the probe tip has been damaged. This damage was caused by an accidental collision with the surface of the specimen stage. Probe tips can become easily damaged because it is difficult to distinguish depths and relative distances between objects using the SEM images. This is mainly a result of the SEM's large depth of field in which 3D objects appear completely in-focus. It is easier to decipher depths when using a light microscope because it has a smaller depth of field which causes 3D objects to appear out-of-focus at varying distances. In addition, the Zyvox nanomanipulator cannot be tilted on the SEM stage because it would collide with the walls of the SEM chamber.

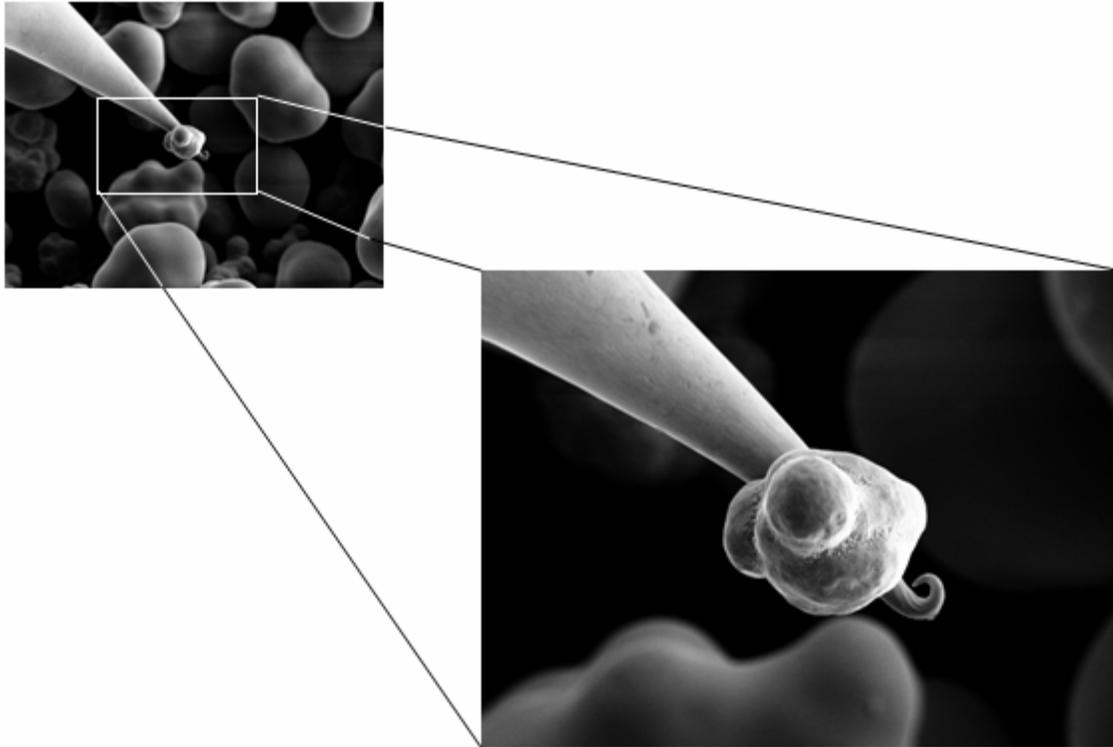


Figure 4.5. Nickel Particle Lifted by Zyvex Probe

Tests were also conducted to evaluate a dielectrophoresis (DEP) grasping model developed by Rohrer et al. (2004). Tests involved applying appropriate voltages to the probe tip using a function generator to create a non-uniform electric field. Ideally, for the DEP model to work, the particle will attach to the probe tip by applying the proper amount of voltage, and it will be released from the probe when the voltage is removed (Rohrer et al., 2004). The grasping behavior through the use of DEP was found to be rather inconsistent and inconclusive. Overall, once particles were attached to the probe, they could not be removed by simply turning off the voltage. This indicates the presence of a residual adhesion force that causes the particles to remain attached to the probe tip. Releasing the attached particles was a difficult task. Particles had to be removed from the probe tip through brute force by bumping them into other particles on the substrate.

Oftentimes particles would become attached to the probe tip even in instances when no voltage was applied. Other times, an applied voltage would not cause particles to attach to the probe tips at all. In the both cases, particles were more likely to attach to the probe tip if the particles had remained underneath the electron beam for over one hour. Thus, it is possible that the particles or probe tips become coated with an insulative material due to EBID. This insulative coating could become charged from prolonged exposure to the electron beam, thus causing electrostatic forces. Figure 4.6 shows the successful grasping and lifting of a particle from a substrate using DEP.

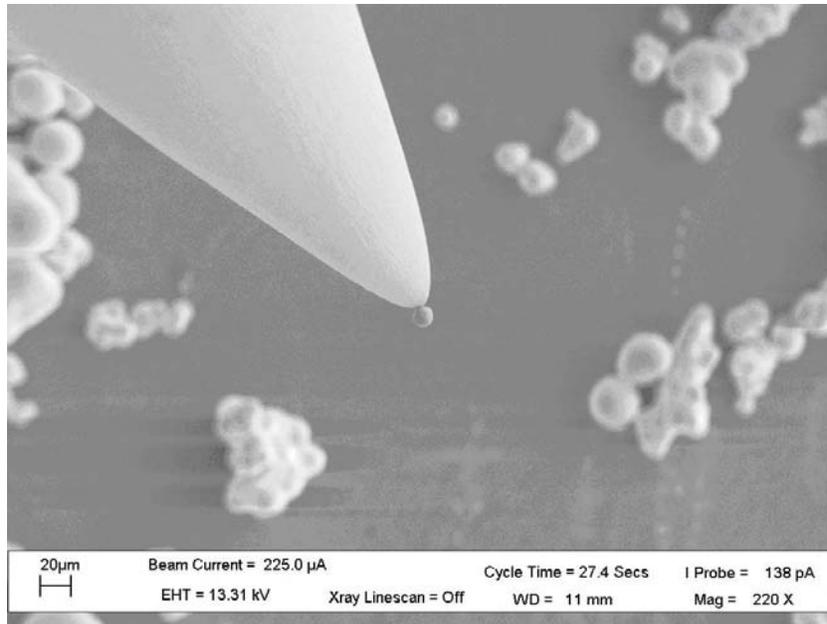


Figure 4.6. Particle Attached to Probe Using DEP

Future work is needed in order to fully evaluate the DEP model and the possible residual attractive forces. Different probe tips and particles made from different types of materials could be investigated. The particles used in these experiments were larger than 10 µm. The dielectrophoretic force may be more effective for smaller particles (1 µm and below). If smaller particles are used, the van der Waals forces on the substrate and the probe tip may also need to be considered.

5. Conclusion

5.1 Summary

This report highlights the findings of an extensive review of the literature in the area of nanorobotics. The main goal of this midyear LDRD effort was to survey and identify accomplishments and advancements that have been made in this relatively new and emerging field. As a result, it may be determined what routes in the area of nanorobotics are scientifically plausible and technically useful so that the Intelligent Systems and Robotics Center can position itself to play a role in the future development of nanotechnology.

There have been extensive publications relating to the field of nanorobotics over the past several years. The bulk of these papers fall into one of the following categories:

- Atomic-scale robots comprised of individually arranged atoms and molecules
- Atomically precise manufacturing which involves the pick and place arrangement of individual atoms to create designer molecules or devices
- Biologically inspired nanorobotics which involves the study of nanorobotic like behavior of agents found in nature
- Microscale robots comprised of nanoscale components
- Manipulation of nanoscale objects using probes with very sharp tips (tip radii of a few nanometers)

Of these categories, atomic-scale robotics and atomically precise manufacturing appear highly speculative. Although there have been some scientific accomplishments such as the writing of the letters “IBM” with individual xenon atoms by Eigler and Schweizer (1990), much of the work to date is purely theoretical (Drexler, 1992; www.imm.org) and many consider it scientifically controversial (Atkinson, 2003). Biologically inspired nanorobotics is a fascinating field of study which will undoubtedly help unlock many of nature’s mysteries. However, biologically inspired nanorobotics is a field currently best suited for study by life scientists, chemists and physicists. Microscale robots comprised of nanoscale components is plausible because many examples exist in nature although scientific progress is slow due to the extreme difficulty involved in fabricating individual components. The manipulation of nanoscale objects using probe microscopes (nanomanipulation) comprises the bulk of published experimental work relating to nanorobotics. Nanomanipulation is interesting because it has provided a mechanism to study the properties of nanomaterials and a process to fabricate prototype nanostructures.

5.2 Recommendations for Future Work

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.

6. References

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