Precision Engineering Division Program

Next-Generation Nanometrology

Solving Industry Needs of Tomorrow

Annual FTEs: 9.5 NIST Staff
8.5 Guest Researchers/Contractors
18 total FTEs

Challenge:

To develop or create new measurement techniques and standards to meet the needs of next-generation advanced manufacturing, which will rely on nanometer scale materials and technologies. The needs for measurement and characterization of new sample structures and characteristics far exceed the capabilities of current measurement science. Anticipated advances in emerging U.S. nanotechnology industries will require revolutionary metrology with higher resolution and accuracy than has previously been envisioned.

Overview

The Next-Generation Nanometrology (NextGen) Program seeks to advance the science of dimensional metrology in order to achieve higher-resolution measurements of increasingly complex samples. This essential research will provide measurement technology in support of emerging U.S nanotechnology industries. The program designs, invents, and develops new metrology instruments for this purpose. Preparation methods for samples and have also become vital research and development challenges, because at these length scales, the sample and the measuring instrument must be treated as integral metrology system. An additional effort is focused on particular challenges presented by certain specific but important advanced measurement needs.

The current general project areas are:

- ***Advanced Optics.*** Although optical microscopy and metrology is a mature field, it continues to see exciting new developments. The NextGen Program has patented and is developing a new technique called scatterfield microscopy that straddles the boundary between optical microscopy (OM) and scatterometry. Significant dimensional information can be extracted from far sub-wavelength features through the use of structured illumination, engineered targets, and physics-based modeling of the instrument and the imaging process. The program is also developing an advanced 193 nm illumination metrology microscope and exploring defect metrology using these methods.

- ***Advanced Particle Beam Metrology.*** The NextGen Program is developing new techniques in various forms of particle beam metrology. In scanning electron microscopy (SEM), those advances include the use of super-sharp emitter tips; active vibration-canceling control of scanned stages; development of standardized resolution-characterization targets and techniques; development of environmental-SEM methods for challenging sample types; and exploration of high-resolution three-dimensional imaging methods. The program is studying the applicability of a dual beam system for metrology problems. The most exciting new advance is the development of a scanning helium ion microscope. The extreme intensity of the single-atom source and the much smaller beam/sample interaction volume permit this technique to obtain higher resolution images than SEM can ever achieve. The different interaction physics of ions rather than electrons also leads to a complementary image contrast mechanism, and holds the potential for nanoscale resolution chemical mapping through energy analysis of emitted and backscattered particles.
- ***Nanoparticle and Materials Metrology.*** The largest existing market for nanotechnology centers on production and use of nanoparticles and nanocomposites. The NextGen Program is developing techniques for fixing soft, bio-mimetic nanoparticles on surfaces without distorting or denaturing them, so that they can be examined with atomic force microscopy (AFM). Medical applications range from imaging contrast agents to therapeutic cancer treatments. Advances in optical tweezers (OT) technology will allow manipulation of particles with sizes reaching down to the nanoscale, giving OT promise as a nanopositioning and nanoassembly tool. Carbon nanotubes, fuel cell membranes, and cellulose single crystals are among the challenging materials for which advanced SEM, OM, and AFM techniques are being investigated.
- ***Atom-based Dimensional Metrology.*** The ultimate limit of nanoscale length metrology is to develop intrinsic calibration standards, where the dimensions are based on integral numbers of atom spacings within an ordered, crystalline lattice. Step height standards have been delivered; linewidth and pitch standards are under development. An ultra-high vacuum scanning tunneling microscope (UHV-STM) plays the major role in these efforts. Validation of atomic lattice spacings as a ruler is being accomplished by comparison with interferometric length measurements in the Molecular Measuring Machine. Extensive work on the development and characterization of STM tips has been an important research area for nanolithography and nanometrology, providing the needed ability to specifically address and modify individual atomic sites.

Key Accomplishments and Impacts:

- The scatterfield microscopy research effort was recognized with the highly respected Nano 50 award for its advances in the new optical technique. Capabilities based on this technique are now being incorporated into commercial products.

- Demonstrated optimized methods for immobilizing intact liposome-based nanoparticle delivery systems (NDS) for scanning probe microscope (SPM) imaging and characterization under fluid conditions. This is a key initial result for enabling physical-property-based production control as opposed to cost-prohibitive biological assays.
- Acquired the first-in-the-world helium ion microscope (HeIM) and began to examine its performance and its capabilities for the metrology of nanoscale objects and features. This is an exciting new imaging technique that has generated significant interest in the nanotechnology community, and may be critical for next-generation semiconductor manufacturing metrology.

Future Directions and Plans:

The NextGen Program is continually seeking new metrology opportunities. The key objective is the pursuit of new dimensional metrology methods, and the application of these advanced measurement techniques to new or emerging measurands that have high value for technological innovation. Cost of ownership and throughput are important considerations as the program seeks to develop and support industrially relevant measurement solutions.

Excellence & Leadership Recognition

Staff	Excellence & Leadership Demonstrated
Silver, Richard Attota, Ravikirran Barnes, Bryan Jun, Jay Stocker, Michael	• Nano 50 Award for Scatterfield Optical Microscopy in the Technologies category for "technology breakthroughs that have, or are expected to have, a significant impact in one or more application areas," 2007
Postek, Michael	• 2007 Nano 50 Award - Nanotech Brief's Nano 50 Awards in the Innovator category "for pioneering achievements in scanning electron microscope imaging and performance improvements and in fostering accurate dimensional metrology and national standards for nanotechnology and nanomanufacturing."
Postek, Michael Villarrubia, John Vladar, Andras	• 2005 Department of Commerce Silver Medal, with John Villarrubia and Andras Vladar for the NIST Model-based Metrology measurement technique and its application to industrial CD metrology.

Projects

Next-Generation Nanometrology Program

Advanced Optics Project

Challenge/Problem Addressed:

The challenge is to advance the state-of-the-art of optical techniques for application to nanometrology problems. We are seeking to obtain dimensional information a factor of ten or more below the wavelength of the light used.

Although optics-based microscopy and metrology are mature fields, exciting new developments continue to arise. The NextGen Program has patented and is developing a new technique called scatterfield microscopy that combines the best features of optical microscopy (OM) and scatterometry. Significant dimensional information with sensitivity to features 1/20th the measurement wavelength can be extracted from the analysis of scattered light profiles through the use of structured illumination, engineered targets, and physics-based image process modeling. Correlated methods have also recently been applied to defect metrology. The project has produced several high impact results for the optical metrology community and the semiconductor industry.

This research effort has led to a number of invention disclosures, patent applications, and publications in leading international journals. Scatterfield techniques are now being developed in other leading optical laboratories and by optical metrology tool manufacturers. Working closely with industrial metrology tool suppliers, we are now implementing these concepts of engineered illumination and target design for super-resolution overlay metrology. Similar success is being achieved in applications of the technique to defect metrology and critical dimension process control and metrology.

This research has been presented at several leading optical technology forums and has helped advance the understanding of high resolution optical measurement applications for semiconductor and nanotechnology manufacturing. This work has demonstrated that the ultimate limits of optical based imaging systems lie far beyond the current state of the art. We have shown that our optical measurement techniques can extend to features 10 nm or smaller in size and that with the appropriate set of techniques could also image very dense features.

Objective(s):

- Develop leading edge optical techniques using the new scatterfield technology. Develop and transfer to industry new optical characterization approaches and methods for optical aberration and illumination control using structured illumination.
- Develop nanometer scale sensitivity using advanced optics techniques for use in next generation linewidth, overlay, and defect metrology. Implement the 193 nm scatterfield optical microscope and evaluate its performance for next generation process control applications.
- Develop advanced optical modeling and instrumentation techniques that can accurately measure sub-40 nanometer sized features or particles for application to the metrology of nanoscale composites and nanotechnology related structures needing high throughput and high resolution manufacturing process control.
- Explore applications of high throughput optical metrology for new applications such as fuel cells, large scale nanoparticles, and patterned defect metrology.

Accomplishments:

- The scatterfield microscopy research effort was recognized with the highly respected Nano 50 award for its advances in the new optical technique. The citation was for the development of a new and revolutionary measurement technique capable of extending conventional optical metrology instrumentation well beyond the current limits. Capabilities based on this technique are now being incorporated into commercial products.
- Patent Applied for on new "Zeroth Order Imaging" method. This new approach combines standard edge-based imaging with signature based methods being developed as part of the scatterfield optical competence project. The key to this approach is to image only the zero order scattered specular reflected light as a function of illumination angle. Features as small as 20 nm in size with a pitch or spacing of 50 nm can be imaged.
- Patent Applied for on new "Super resolution overlay targets". Joint NIST/SEMATECH patent has been formally applied for new super-resolution overlay target. Leading optical instrument manufacturers are very interested. These targets have the potential to change the target designs and methodology widely used by industry.
- The new 365 nm illumination scatterfield microscope has been completed. This new tool is being developed from the ground up at NIST specifically for the purpose of using illumination engineering and structured target designs. The centerpiece of the microscope is the open architecture of the illumination path, which features a breadboard-based design. This design will allow us to investigate quickly methods for engineering the illumination to probe the scattered field.
- Obtained SEMATECH contract for an investigation to the fundamental limits of optical critical dimension measurement techniques. Although largely focused on scatterometry techniques, the world-class optical modeling capabilities at NIST are now being applied to scatterfield and scatterometry optical methods.
- A recent document titled "Summary of 2006 NIST/SEMATECH Studies of Next Generation Overlay Metrology," prepared and authored by SEMATECH, has highlighted a number of recent advances by staff in the scatterfield competence project. The report goes into detail on collaborations between NIST optical researchers, SEMATECH staff, and the semiconductor industry in the overlay metrology area.

Planned Future Accomplishments:

- Complete the 193 nm optical tool platform. Demonstrate full operation in a cleanroom environment with controlled temperature. (Complete by 2nd Quarter 2009)
- Develop test patterns based on optical superstructures with applications in nanotechnology process control. (Complete by 4th Quarter 2009)
- Demonstrate the highest resolution optical measurements using scanned aperture methods for dimensional metrology of sub-15 nm sized features with measurement sensitivity significantly better than 1 nm. (Complete by 4th Quarter 2010)
- Test and implement a new class of structured illumination using automated, model-based pupil engineering with LCD or DLP back focal plane engineered illumination. (Complete by 4th Quarter 2011)

Customers and Collaborators:

- KLA-Tencor, Nanometrics and Applied Materials
- SEMATECH
- IBM, Intel, AMD, TI

Advanced Particle Beam Metrology Project

Challenge/Problem Addressed:

Continuing progress in nanotechnology requires parallel improvement in measurement resolution and greater versatility in the quantities – or measurands – that can be examined. The challenge is to create new strategies and measurement techniques that go beyond those currently available. This project focuses on this challenge within the realm of particle beam imaging.

Scanning electron microscopy (SEM) is one of the workhorse techniques for imaging and characterization for nanotechnology. We are working to develop advanced methods to enhance its capabilities. We are developing sample manipulation stages with high-speed, sub-nanometer-resolution laser interferometer measurement that allow active monitoring or control of stage vibration, a major contributor to image blurring. Research is also being done on the development of super-tips for the electron emitters, to make a brighter source that will increase resolution. Significant contributions to the advancement of SEM have also come through the development of resolution standards and resolution measurement methods, which places the characterization of this most critical of SEM performance metrics on a solid metrological basis and allows objective comparison between instruments and operating conditions. New SEM tools under development include an environmental SEM (e-SEM) that allows imaging of uncoated insulating materials. We are also developing the metrology applications of a dual beam system that has a focused ion beam (FIB) for ion milling along with an SEM for concurrent imaging. This instrument allows the localized, in-situ, cross-sectioning of features of interest for 3-dimensional metrology.

The most exciting recent advance in particle beam metrology is the development of a new technique called scanning helium ion microscopy (HeIM), which is analogous SEM but uses a probing beam of He^+ ions instead of electrons. MEL owns the world's first commercial HeIM and is working closely with the manufacturer to understand the science of He^+ ion imaging and to optimize the instrument's metrological capabilities. HeIM results so far show competitive or better imaging capabilities than the most advanced SEMs. The sample interaction volume for He^+ ions is significantly smaller than for electrons, leading to higher resolution. The interaction physics is also different, leading to a different contrast mechanism and the extraction of complementary information.

Objective(s):

- Develop instrumental improvements for particle-beam microscopy to enable higher resolution images with less noise.
- Develop standard methods, standard data sets, and calibration artifacts for quantifying the imaging resolution of particle beam microscopes.
- Explore the range of measurands that might be accessible with a He^+ ion beam scanning microscope, such as nanometer-scale compositional analysis.

Accomplishments:

- Developed a SEM resolution reference material and associated algorithms for the objective characterization of SEM performance. This will allow a fair comparison between competing instruments and operating conditions.
- Developed reference simulated image data sets with known added noise types for the objective characterization and comparison of image resolution software.
- Demonstrated the acquisition of high speed stage position data correlated with secondary electron intensity. This allows sorting of the image data based on actual stage position, leading to a significant reduction in image noise.
- Developed a method to measure the 3D electron beam profile in an SEM by using thin layers of photoresist on silicon chips and exposing them using various focus settings, irradiation time, and beam current. This provides a solid experimental basis to validate the modeling of the interaction volume for electron beam exposure of specimens, leading to more accurate metrology.
- Developed new SEM image modeling software capable of performing simulations for fully 3-dimensional samples. This capability is critical for the new generation of semiconductor devices being developed in industry.

Planned Future Accomplishments:

- Reduce vibration noise effects on SEM imaging by developing real-time image data sorting capability based on the vibration data acquired by the laser stage. (Complete by 4th Quarter 2010)
- Characterize the sensitivity of the HeIM for high-resolution metrology of linewidths in silicon, photoresist, and mask materials. (Complete by 4th Quarter 2009)
- Model ion transport, ion energy loss, and secondary electron production by ions in samples. This will provide us with a metrology capability for accurate measurements with the helium ion microscope, which has the promise of higher spatial resolution than the SEM and therefore much promise as a CD metrology tool. (Complete by 4th Quarter 2013) (Initial model by 4th Quarter 2011)

Customers and Collaborators:

- Zeiss
- FEI
- Hitachi
- E. Fjeld

Nanoparticle and Materials Metrology Project

Challenge/Problem Addressed:

Nanoparticles and materials containing nanoparticles represent by far the largest fraction of the current nanotechnology product market. Yet the methods available for characterization and dimensional measurement of these products are inadequate for efficient development and cost effective production. The challenge is to find reliable, repeatable, and cost effective methods for measuring nanoparticles and nanoparticle-based materials, and to provide the infrastructure needed for measurement traceability. Special challenges include the imaging of soft, deformable materials, and materials with limited sample homogeneity.

For nanoparticle metrology, we are developing measurement approaches that address the challenges of measuring soft particles. For medical applications, for example, we are developing a systematic methodology for directly determining physical and biological information about nanoparticle delivery systems (NDS) from image data. This approach uses fluid scanning probe microscopy (SPM) as a versatile tool for high-resolution imaging of biological systems in solution. Fluid SPM refers to the operation of a scanning probe microscope under solution conditions. It includes the development of specialized techniques for sample preparation, stable feedback operation of an oscillating probe tip in solution, and quantitative force measurements of chemical, mechanical, electrical, hydrodynamic, and magnetic interactions of the molecular constituents of an NDS. Appropriate preparation and measurement techniques for physical characterization of nanobioparticles have been developed in parallel with *in-vitro* and *in-vivo* efficacy studies in order to facilitate drug discovery and development. These studies were done in collaboration with the Georgetown University Medical Center (GUMC).

We are developing an optical tweezers system for the trapping and manipulation of nanoparticles. This will make possible the controlled placement of nanoparticles at predefined locations on substrates. Simultaneous trapping of multiple particles and control of the orientation of non-symmetric nanoparticles have been demonstrated. This instrument will be exercised as a test-bed for nanomanufacturing and assembly. More immediately, the controlled placement of nanoparticles will permit an ordered-array standard reference material (SRM) for nanoparticle dimensions.

The applicability of SEM, SPM and advanced optical methods for the characterization of several advanced materials, including carbon nanotubes, cellulose single crystals, and fuel cell membranes is also being examined.

Objective(s):

- Develop sample preparation techniques and instrumental improvements for accurate nanoparticle characterization and metrology.
- Develop standard methods, standard data sets, and calibration artifacts for quantifying the imaging and metrology of nanoparticles.

Accomplishments:

- Demonstrated optimized methods for immobilizing intact liposome-based nanoparticle delivery systems (NDS) for scanning probe microscope (SPM) imaging and characterization under fluid conditions. This is a key initial result for enabling physical-property-based production control as opposed to cost-prohibitive biological assays.
- Fabricated and evaluated improved magnetic coatings for fluid and dry magnetic force microscopy (MFM) for characterizing dispersed and aggregated contrast enhancement agents for magnetic resonance imaging (MRI).
- Investigated quantitative particle size analysis of gold reference nanoparticles, NDS, and superparamagnetic iron oxide nanoparticles (SPIOs) from SPM images, and compared with results from dynamic light scattering (DLS).
- Measured the size and shape of the soon-to-be-released Colloidal Gold Nano Particle Reference Materials (RM 8011, RM 8012 and RM 8013) using a reference SEM.
- Demonstrated simultaneous optical trapping and individual manipulation of multiple particles.
- Measured carbon nanotubes samples with SEM for use as a carbon nanotubes (CNT) reference material.

Planned Future Accomplishments:

- Fully implement a new technique, the rotating disk method for measuring surface zeta potentials of substrates, in order to achieve optimization of nonspecific surface binding for nanoparticle delivery systems, an essential first step in high-magnification SPM characterization. (Complete by 4th Quarter 2011)
- Apply novel preparation methods to superparamagnetic iron oxide (SPIO) nanoparticles for thorough magnetic-based characterization of the particles under dispersed and aggregated conditions. SPIOs have become a significant contrast agent for biomedical research. (Complete by 4th Quarter 2010)
- Demonstrate active, high-bandwidth control of optical trap intensity and position to enable the trapping of 200 nm diameter or smaller nanoparticles. (Complete by 4th Quarter 2011)
- Demonstrate the controlled placement of nanoparticles on a substrate for the development of a structured nanoparticle dimensional reference standard. (Complete by 4th Quarter 2010)

Customers and Collaborators:

- Georgetown University Medical Center (GUMC)
- University of Akron
- National Cancer Institute (NCI) Nanotechnology Characterization Laboratory (NCL)

Atom-based Dimensional Metrology Project

Challenge/Problem Addressed:

The ultimate future for nanoscale dimensional metrology is to develop intrinsic standards based on integral numbers of atom spacings within an ordered, crystalline lattice. Single-atom step height standards have been fabricated and calibrated; linewidth and pitch standards are under development. Instrumentation requirements are very challenging, typically requiring an ultra-high vacuum environment scanning tunneling microscopes (UHV-STM). STM tip creation technology is an important aspect of this work, since nanolithography and nanometrology instruments must routinely operate with atomic-dimension resolution.

In this work we are developing a new, comprehensive approach to dimensional metrology using the atom spacings of a crystal as the underlying “ruler” or scale. Based on this metric, larger scale features are etched in the surface to serve as critical dimension or pitch references for instruments having less-than-atomic resolution.

This project has produced several important technical results, some of which have recently been published in Applied Physics Letters, Optical Engineering, and the Journal of Chemical Physics. Technical outputs include 20 pm resolution interferometry, chemically-prepared atomically-flat silicon substrates, atomic lattice-based metrology methods, 10 nm lithography, field-ion/field-electron microscope (FIFEM) and STM tip development, silicon step-flow dynamics simulations, and nanometrology test target fabrication.

Two complex multi chamber vacuum STM imaging facilities are used in this effort. The first system is a five chamber ultra high vacuum (UHV) facility regularly capable of producing 133 x 10^{-10} Pa (1 x 10^{-10} torr) base pressures, along with vibration isolation that yields a sub-angstrom noise floor. The other system has an FIFEM, which is used to image scanned probe tips on the atomic scale; this has become an essential tool for atomic resolution work as well as more recent linewidth metrology work. This system also has an STM equipped with UHV interferometry in the first application of a direct atomic imaging system with interferometry.

A second aspect of this project is the continued development of improved nanofabrication methods using the STM. A key focus in this work is the use of improved wet chemistry methods for etching and hydrogen termination of silicon substrates, hard etch mask formation using the STM, and their subsequent reactive ion etching. This research involves the writing of features on silicon substrates in hydrogen terminated substrates. The removal of hydrogen atoms and exploration of ways to improve and reduce the feature size is crucial to advancing this nanofabrication process. The hydrogen terminated written features are then processed with our newly developed reactive ion etching (RIE) process.

The project is investigating new methods for high resolution STM imaging using alternative tip materials. We have used the FIFEM to image tungsten STM tips (field emitters) and have also subsequently attached nanotubes to them.

Objective(s):

- Develop atom-based fabrication techniques for the sub-5 nm to atomic scale critical dimension domain. Use the hard etch mask oxidation technique with scanning tunneling microscopy on Si (111) and Si (100) surfaces.
- Evaluate new tip technologies for robustness based FIFEM and atomic imaging capabilities. Work closely with industry and DARPA partners to obtain state-of-the-art tips and evaluate their use with our industrial and university partners.
- Develop specific preparation and imaging requirements to determine dimensions based on the crystal lattice. Implement atom-based dimensional metrology techniques directly linked to the crystal lattice. These will be developed using STM, AFM, and transmission electron microscope (TEM) techniques as appropriate.

Accomplishments:

- Two leading publications in the Journal of Chemical Physics that present a detailed theoretical and experimental understanding of the preparation of Si (111) surfaces.
- Comprehensive collaborations with the University of Maryland and George Washington University have resulted in numerous publications covering much of the custom designed hardware.
- Recent collaborations with leading university and nanotechnology manufacturing concerns have recently evolved to a long term funded structure aimed at commercialization of advanced single and multi probe metrology and fabrication techniques.

Planned Future Accomplishments:

- Develop techniques for improved field emission microscopy. Image W(111) and alternative tip materials and evaluate their atomic resolution imaging capabilities. (Complete by 4th Quarter 2011)
- Develop wet silicon processing methods to enable surface and atomic reconstruction on silicon etched structures at room temperature. (Completed by 4th Quarter 2010)
- Demonstrate new methods for nanofabrication of silicon based structures on the nanometer scale. This includes the investigation of Si (100) for enhanced and repeatable nanomanufacturing of features with sub-10 nm dimensions. (Completed by 4th Quarter 2010)

Customers and Collaborators:

- George Washington University
- University of Maryland, Depts. of Physics and Chemical Physics Program
- Zyvex and University of Illinois