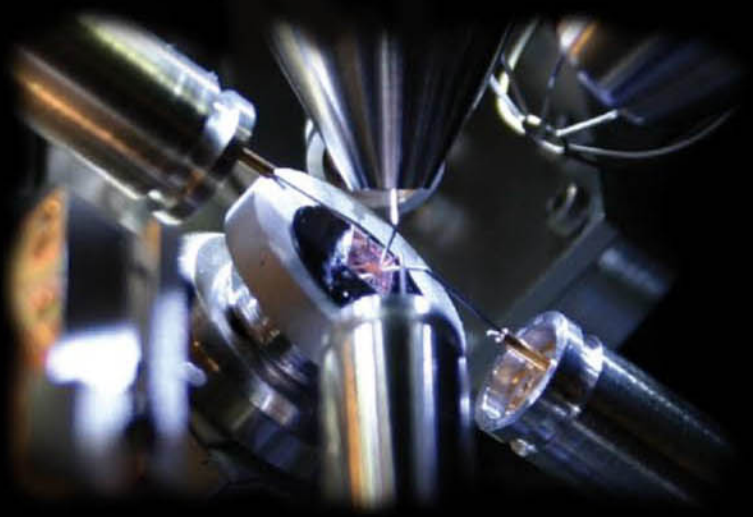
Instrumentation and Metrology for Nanotechnology

Report of the
National Nanotechnology Initiative Workshop
January 27-29, 2004



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The Nanoscale Science, Engineering, and Technology (NSET) Subcommittee is the interagency body responsible for coordinating, planning, implementing, and reviewing the National Nanotechnology Initiative. NSET is a subcommittee of the National Science and Technology Council (NSTC), which is one of the principal means by which the President coordinates science, space, and technology policies across the Federal Government. The National Nanotechnology Coordination Office (NNCO) provides technical and administrative support to the NSET Subcommittee and supports the subcommittee in the preparation of multi-agency planning, budget, and assessment documents, including this report.

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For more information on NSTC, see http://www.ostp.gov/nstc/.

For more information on the NNI, NSET, and NNCO, see http://www.nano.gov.

About this document

This document is the report of a workshop held under NSET auspices in January 2004 seeking input from the research community on the NNI research agenda relating to capabilities needed for instrumentation and metrology for nanotechnology. It was used as input for the NNI Strategic Plan released in December 2004. The meeting was jointly sponsored by the National Institute of Standards and Technology, Technology Administration, U.S. Department of Commerce, and, through the NNCO, the other member agencies of the NSET Subcommittee.

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Back cover: Digital Instruments multimode SPM vector piezoresponse force microscopy image of lead titanate thin film (courtesy of A. Gruverman and B.J. Rodriguez, North Carolina State University, and S.V. Kalinin, J. Shin and A. Baddorf, Oak Ridge National Laboratory).

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Instrumentation and Metrology for Nanotechnology

Report of the National Nanotechnology Initiative Workshop
January 27–29, 2004, Gaithersburg, MD

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Sponsored by

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National Institute of Standards and Technology
Technology Administration
U.S. Department of Commerce

ACKNOWLEDGMENTS

Thanks go to the principal authors of this report, who are listed on the title pages of the respective chapters. In addition, we thank all participants of the January 27–29, 2004 NNI Interagency Workshop on Instrumentation and Metrology for Nanotechnology Grand Challenges, held at the National Institute of Standards and Technology (NIST), an agency of the U.S. Department of Commerce's Technology Administration, in Gaithersburg, Maryland. A complete list of attendees is provided in Appendix B. The participants braved inclement weather (freezing rain, snow, and sleet) to attend this workshop. Special thanks are extended to the members of the workshop technical organizing committee, listed below.

The presentations and discussions that took place at the workshop provided the foundation for this report. Without this valuable input this report would not have been possible. Many thanks are also due to the conference support staff of NIST, especially Kathy Kilmer, Angela Ellis, Dean Smith, Sarah Bell, and Ken Osbourne, who made sure that the meeting logistics were handled properly and efficiently.

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Finally, thanks to all the members of the National Science and Technology Council's Subcommittee on Nanoscale Science, Engineering, and Technology, who through the National Nanotechnology Coordination Office, cosponsored the workshop with NIST and reviewed the draft report before publication.

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The workshop was sponsored by the National Institute of Standards and Technology, an agency of the U.S. Department of Commerce's Technology Administration and, through the National Nanotechnology Coordination Office (NNCO), the other member agencies of the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee, Committee on Technology, National Science and Technology Council. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the United States Government or the authors' parent institutions.

PREFACE

This report on Instrumentation and Metrology for Nanotechnology is one of a series of reports resulting from topical workshops convened during 2003 and 2004 by the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council's Committee on Technology through the National Nanotechnology Coordination Office (NNCO). The workshops were part of the NSET Subcommittee's long-range planning effort for the National Nanotechnology Initiative (NNI), the multiagency Federal nanotechnology program. The NNI is driven by long-term goals based on broad community input, in part received through these workshops. The NNI seeks to accelerate the research, development, and deployment of nanotechnology to address national needs, enhance our nation's economy, and improve the quality of life in the United States and around the world, through coordination of activities and programs across the Federal Government.

At each of the topical workshops, nanotechnology experts from industry, academia and government were asked to develop broad, long-term (ten years or longer), visionary goals and to identify scientific and technological barriers that once overcome will enable advances toward those goals. The reports resulting from this series of workshops inform the respective professional communities, as well as various organizations that have responsibilities for coordinating, implementing, and guiding the NNI. The reports also provide direction to researchers and program managers in specific areas of nanotechnology R&D regarding long-term goals and hard problems.

This report is the result of a workshop held under NSET Subcommittee auspices in January 2004 seeking input from the research community on the NNI research agenda related to one of the original NNI "grand challenge" topics, "Nanoscale Instrumentation and Metrology." The findings from this workshop were used in formulating the new NNI Strategic Plan released in December 2004, particularly the Program Component Area (PCA) on Instrumentation Research, Metrology, and Standards for Nanotechnology described in that plan. This report will continue to inform the NNI research program under that PCA. The report was also co-sponsored by the National Institute of Standards and Technology (NIST) to provide input to its research agenda within the overall NNI program.

The report identifies and highlights research needs in five priority areas for nanotechnology-related instrumentation and metrology: (1) nanocharacterization; (2) nanomechanics; (3) nanoelectronics, nanomagnetics, and nanophotonics; (4) nanofabrication; and (5) nanomanufacturing. It also includes a discussion of crosscutting computational science issues and challenges.

On behalf of the NSET Subcommittee, we wish to thank Dr. Michael Postek of NIST and Prof. Robert Hocken of the University of North Carolina for their creativity and leadership in conducting an outstanding workshop and in preparing this report, as well as all the other NIST staff members for their hard work in organizing the workshop and report. We also thank all the speakers, session chairs, and participants for their individual contributions to the discussions at the workshop and to the drafting of this report. Their generous sharing of the results of their research and their insights ensures that this document will serve as a valuable reference for the NNI.

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EXECUTIVE SUMMARY

INTRODUCTION

Instrumentation and metrology are both integral to the emerging nanotechnology enterprise, and have been identified by the National Nanotechnology Initiative (NNI) as critical nanotechnology areas. Instrumentation and metrology are vital to applications in everything from electronics to medicine and crosscut all the NNI areas of research and application. Advances in fundamental nanoscience, design of new nanomaterials, and ultimately manufacturing of new nanotechnology-based products will all depend to some degree on the capability to accurately and reproducibly measure properties and performance characteristics at the nanoscale.

Advances in instrumentation and metrology have enabled two decades of remarkable nanoscience and nanotechnology research. However, the resolution, accuracy, and capability of currently available instruments and tools are being stretched to the limit by the demands of researchers and are not expected to meet many of the needs posed by those seeking to incorporate nanotechnology into commercial products and manufacturing processes. To meet some requirements, revolutionary rather than evolutionary advances will be needed.

To gain input from stakeholders on the nanoscale metrology capabilities that will be needed in future, the NNI Interagency Workshop on Instrumentation and Metrology for Nanotechnology Grand Challenges was held on January 27–29, 2004 in Gaithersburg, Maryland, and was cosponsored by the National Institute of Standards and Technology (NIST), an agency of the U.S. Department of Commerce's Technology Administration, and the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council. Over 200 nanotechnology experts from industry, academe, Federal agencies, and private research institutes attended the workshop. Participants provided insights on industry metrology needs and the research and development (R&D) that should be undertaken to develop the necessary instrumentation and metrology capabilities.

Breakout Session Focus Topics

- Nanocharacterization (physical and chemical properties, structures)
- Nanomechanics (mechanical properties, tribology)
- Nanoelectronics, nanomagnetics, and nanophotonics (device performance and materials properties)
- Nanofabrication (instrumentation for nanofabricated structures, devices)
- Nanomanufacturing (mass production, fast measurement technology for production applications)

This report summarizes the important outcomes of the workshop. The report is organized around five topics (see sidebar at left) and includes for each topic an overview of the current state of the art, goals and challenges, a vision for the future, recommendations for future research, and implementation strategies to accelerate the development of important technology. In addition, a summary of crosscutting computational science issues and challenges is treated as an additional topic. Although not a central focus of the workshop, computational science was recognized as an important element in the future success of the nanometrology component of the NNI. Appendices to the report include the workshop agenda, a list of workshop participants, abstracts submitted in advance of the workshop, and a glossary.

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

Entirely new metrology tools will be required to meet the needs of emerging nanotechnologies. Currently available equipment in most cases is at the limits of resolution, and much greater metrology capabilities will be required for every area from laboratory research to commercial-scale manufacturing.

Although instrumentation developed for use in existing industries, such as the manufacture of semiconductors, catalysts, and chemicals, has some application, it may be of limited usefulness in other nanotechnology-related industries. The Chemical Industry R&D Roadmap for Nanomaterials by Design (http://www.ChemicalVision2020.org) emphasizes metrology as a key enabling technology for the discovery, development, and manufacture of emerging nanomaterials and systems, citing specific needs in real-time analytical and characterization tools, standardization, and informatics. Even industries that have led development of tools for manufacturing at the nanoscale anticipate impending needs. For example, the International Technology Roadmap for Semiconductors (ITRS) reports that in 5–10 years, no known solutions will be available for many critical metrology tasks, making fundamental metrology research today a must.

Funding is needed to build up educational programs, upgrade university facilities, and create a multidisciplinary research community for metrology at the nanoscale. Education and training opportunities should be pursued to create the skilled workforce needed to support nanotechnology R&D from basic characterization of properties to fabrication and manufacturing.

IMPLEMENTATION STRATEGIES

Successful implementation of this report's recommendations will depend on effective collaboration and cost-sharing between a diversity of Federal agencies and industry stakeholders. Federal laboratories and universities will play an important role in the development of new metrology tools; all stakeholders should be involved in experimental validation, testing, and demonstration for commercial application. Open-access technology centers (user facilities) at universities or Federal laboratories should be used to provide expertise and capabilities.

Key strategies for the NNI and the research community going forward include continuing interaction (e.g., domestic and international workshops) with potential partners and stakeholders to refine and guide the R&D pathway, evaluate progress toward milestones established by the NSET Subcommittee, and identify critical funding shortfalls. The development of technology-specific roadmaps that further define future R&D activities could emerge from these interactions. The development of a national (or international) technology roadmap for nanotechnology (NTRN) for instrumentation and metrology similar to the current ITRS is another challenge for the research community to consider. Such a roadmap for instrumentation and metrology would not only guide technology development, but also guide instrument manufacturers to provide the needed tools with reasonable lead time. Instrument development associated with the semiconductor manufacturing industry was an evolutionary process fueled by the defined needs of the ITRS and funded by the established semiconductor industry. The emerging nanomanufacturing industry does not have sufficiently deep pockets to fund similar high-risk development, and this creates a significant funding gap. A significant challenge is identifying and establishing funding sources for the high-risk development of a diversity of needed instrumentation, some of which may need to be revolutionary.

Technology development and demonstration should be conducted with a close eye on regulatory issues, and regulatory agencies should be included in stakeholder interactions, as appropriate.

Sustainability issues, in parallel with health and safety issues, should be considered as development of technology progresses.

Nanotechnology currently spans across and is represented in varied industries. If nanotechnological commonalities and focus could be derived, a consortium-type organization or organizations initially co-funded by government and industry could make huge strides in needed instrumentation and metrology and act as the focal point for roadmaps. Participating agencies (e.g., Department of Commerce, Department of Defense, Department of Energy, National Science Foundation, National Institutes of Health, and the Department of Agriculture) could map their program areas to align with the diverse potential nanotechnology application areas to accomplish targets set by the roadmaps.

Probing nanoscale devices and systems will require revolutionary developments in addition to evolutionary advances in measurement schemes and devices. Scientists in academia, government laboratories, and industry need to focus on instrument research and development, and increased funding should be directed into this area. In addition, programs that would encourage the large equipment manufacturers interested in development of new equipment should be enhanced. Another issue that workshop participants raised is whether the current workforce has the skill sets needed to develop new tools. Strategies must include building up the necessary education infrastructure and technical knowledge base.

RECOMMENDATIONS

Each of the chapters of this report includes recommendations specific to the individual breakout session topics covered at the workshop. The overarching grand challenge derived from the workshop, which essentially summarizes all the individual challenges for the areas surveyed, is *to develop the ability to determine the elemental composition, location, and chemical state of all atoms in a nanostructure in three dimensions with atomic accuracy, and the ability to understand and predict the resulting properties of the nanostructure.* This requires the development of new metrology instrumentation and infrastructure for both laboratory research and nanomanufacturing. Broad-based recommendations to develop the instrumentation and metrology required to enable nanotechnology and the future manufacturing of nanotechnology-based products are:

- Develop a national (or international) technology roadmap for nanotechnology for instrumentation and metrology similar to the current International Technology Roadmap for Semiconductors to guide technology development and assist instrument manufacturers in providing measurement tools within a reasonable lead time
- Develop strong educational programs and leverage Federal laboratories that address the development of measurement infrastructure and advanced measurement instrumentation
- Coordinate funding of educational programs with agencies to provide effective support for program areas of joint interest
- Leverage national laboratories' user facilities to foster the development of new measurement techniques and development of a national user facility for nanometrology
- Foster the development of consortia cofunded by government and industry tasked to bridge the gap for the development of sector-specific instrumentation for nanometrology for nanomanufacturing
- Invest in integrated computational methods to develop predictive and assessment tools for nanometrology and nanomanufacturing

1. INTRODUCTION

Michael Postek, NIST

BACKGROUND

Nanotechnology involves the manipulation of matter at the nanometer length scale to create nanostructures with unique properties. Through nanotechnology, it is envisioned that a dazzling array of new materials, devices and products can be made possible, improving our quality of life and generating positive economic and societal effects.

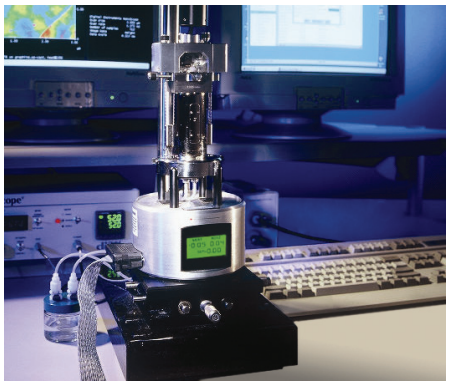

Figure 1.1. MultiMode Atomic Force Microscope for imaging small samples at high resolution (courtesy of Veeco Instruments).

Instrumentation and metrology (the science of measurement) is a key underpinning of the emerging nanotechnology enterprise. Advances in fundamental nanoscience and ultimately manufacturing of new nanotechnology-based products will all depend to a great degree on our capability to measure accurately and reproducibly properties and performance characteristics at the nanometer scale.

New nanotechnology-based industries that mass-produce products will require high-performance, cost-effective, reliable instrumentation and improved measurement methods (metrology). Along with these requirements comes the need for effective collection, transmission, and interpretation of measurement information and data. As new nanostructures are fabricated, assembled, and manufactured into usable products, standardized instrumentation and metrology will be vital for providing quality control and ensuring reproducible performance. Globally accepted standards for measurement and identification of properties and structures at the nanoscale will be necessary for ensuring that U.S. products compete in the international marketplace.

Selected Properties Measured at the Nanoscale

Physical and chemical properties—force, strength, length, chemical composition, shapes of pores and particles, form, surface area, surface topography and sub-surface damage

Mechanical—elasticity, hardness, friction, adhesion, durability

Electronic, photonic and magnetic properties on surface or buried/embedded—resistance, dielectric constant, refractive index, emissivity, hysteresis, domains, spin tunneling femtosecond measurements

Fabrication and manufacturing—structures, surface interactions, internal features, dynamics of assembly

Decades of nanoscience research have led to remarkable progress in nanotechnology as well as an evolution of instrumentation and metrology suitable for some nanoscale measurements. The currently available suite of metrology tools is capable of meeting the needs of exploratory nanoscale research. However, as viable nanoscale applications emerge, new techniques, tools, instruments, and infrastructure will be needed to support further research. In addition, manufacturing metrology and instrumentation must be in place for

successful manufacturing of nanotechnology-based products on a commercial scale. Two previous industrial revolutions—the machine revolution at the end of the 19th century and beginning of the last century and the semiconductor revolution in the middle of the 20th century—demonstrate that metrology has been key to enabling the widespread adoption of new technology. The same can be said of the developing nanotechnology revolution.

Accurate measurement of dimensions, characterization of materials, and elucidation of structures at the nanoscale are critical, from exploratory research to concept and prototyping and ultimately manufacturing. While metrology tools exist today, they are reaching limitations for resolution, accuracy, and capability at the nanoscale, and will not meet future requirements for nanotechnology-based products.

A better understanding of the life-cycle implications of nanotechnology for the environment and human health and safety will also rely to a large degree on the measurement of properties at the nanoscale. Today, such measurements present a significant challenge for all aspects of metrology. Advanced measurement science will be necessary, for example, to detect trace levels from exposure to nanomaterials resulting from medical, occupational, environmental, or accidental release. Hence, it will be essential to develop the instrumentation and metrology to accurately follow the environmental fate of nanoscale materials, develop safe nanoscale sample handling methods, and accurately measure the effects.

THE WORKSHOP

To address the issues outlined above and seek input from the research community, the National Nanotechnology Initiative (NNI) Interagency Workshop on Instrumentation and Metrology for Nanotechnology Grand Challenges was convened on January 27–29, 2004 in Gaithersburg, Maryland. The workshop was sponsored by the National Institute of Standards and Technology (NIST), an agency of the U.S. Department of Commerce's Technology Administration, and the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council's Committee on Technology. The agenda is included as Appendix A to this report. The workshop was attended by over 200 nanotechnology experts from industry, academe, Federal agencies, and private research institutes (see Appendix B).

The plenary session included keynote presentations with remarks from top Federal officials and visionary presentations from researchers in the field as well as from firms developing nanotechnologies. The initial presentations were followed by a day and a half of focused breakout sessions on instrumentation and metrology for the following five topics:

1. *Nanocharacterization:* measurement of physical and chemical properties such as dimension/size, force, composition, surface area, and shape of nanoscale materials and devices; includes imaging of the three-dimensional (3D) relationships of complex nanoscale components

2. *Nanomechanics:* measurement of the mechanical properties such as friction, hardness, elasticity, adhesion, durability, and performance of nanostructured materials in devices and systems; includes nanoindentation and nanotribology as applied to the mechanics of constrained volume materials

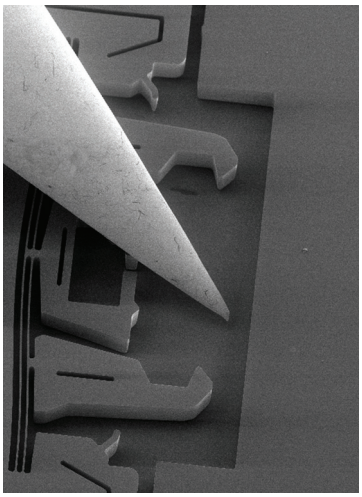

Figure 1.2. Zyvex NanoEffector® Probe on a MEMS component (courtesy of Zyvex Corporation).

3. *Nanoelectronics, nanophotonics, and nanomagnetics:* reproducible measurement of electronic, photonic or magnetic properties (surface or embedded) such as resistance, refractive index, emissivity, of nanoscale devices and materials as needed to successfully incorporate devices into commercial products

4. *Nanofabrication:* metrology to support fabrication of device-like structures with features having dimensions as small as a single atom; includes manipulation and placement of individual atoms and molecules, and external instrumentation to interact with structures and devices

5. *Nanomanufacturing:* measurement methods to support the mass manufacture of nanotechnology-based products; includes the ability to measure, control, and predict the nanoscale structure, performance, and properties of materials and devices over millimeter scales reliably, reproducibly, and on the production floor

An additional special session was held to assess the computational science needs common to the other focus areas. Each breakout session included plenary presentations to provide perspective on the state of the art from key researchers. In addition, facilitated sessions were held to discuss visionary goals for each area, prioritize future needs, and identify the key technical barriers and challenges.

From each breakout session emerged a set of prioritized challenges that are expected to form the components of a set of grand challenges for nanoscale instrumentation and metrology.

THE REPORT

The remainder of this report outlines the technology challenges and research needs unique to each of the areas outlined above. The report presents the ideas that were generated by the various breakout groups and recommendations for future research and development. Reports from other workshops provided added background and context. Abstracts submitted by participants in advance of the workshop were another important source of information and are included in Appendix C of the report. A glossary is included as Appendix D.

The objective of the workshop and this report is to provide guidance on the research and development priorities for future nanoscale instrumentation and metrology. The report includes an overview of the current state of the art, goals and challenges, a vision for the future, recommendations for future research in nanoscale metrology, and implementation strategies to accelerate the development of important technology.

2. Instrumentation and Metrology for Nanocharacterization

Principal Contributing Author: Richard Cavanagh

SCOPE

Nanocharacterization spans issues in physical and chemical metrology, including force and length measurements, chemical composition determination, shapes of pores and particles, and 3D relationships of complex nanoscale components. To support the emerging nanotechnology industry, advances in nanocharacterization will be required, including:

- Development of appropriate measurement expertise
- Realization of nanoscale 3D imaging capabilities
- Acquisition of measurement methods that are scientifically sound and artifact free
- Availability of quantitative metrology with analytical capabilities that parallel what is currently achievable on the microscale

A combination of measurement capabilities will be needed to address nanocharacterization challenges. These either will extend existing measurement techniques such as those used by microscopists and spectroscopists, or will emerge from the invention of new measurement methods that enable both compositional and performance factors to be quantitatively and reproducibly measured on the nanoscale.

VISION

Characterization should also be done *in situ*, in the sample's natural environment, if possible. Techniques should provide both the basis for understanding and leveraging measurement results from bulk techniques and tools that measure collective systems and distributions in a statistical fashion and support a global measurement infrastructure based on intercomparability and multimodal compatibility.

Vision for Nanocharacterization

The vision for the future is to achieve advanced methods and metrology techniques to characterize complex, heterogeneous samples (organic and inorganic systems) in three dimensions over all relevant time and length scales (e.g., with 1 nm spatial resolution).

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

The 1986 Nobel Prize in Physics was shared by Ernst Ruska for his work in electron microscopy and by Heinrich Rohrer and Gerd Binnig for their discovery of the scanning tunneling microscope [1-3]. The intervening years have witnessed an unparalleled growth in our ability to characterize structures and complex materials at ever-increasing spatial resolution.

Electron microscopies (scanning electron microscopy—SEM—and transmission electron microscopy—TEM), proximal probe microscopies such as scanning tunneling microscopy (STM), atomic force microscopy (AFM), and to a lesser extent near-field scanning optical microscopy (NSOM), are currently the tools of choice for nanoscience and nanotechnology research [4, 5]. These microscopies provide researchers with the ability to image the samples and view sample surfaces on the atomic scale as well as with the ability to *manipulate* the particles (atoms, molecules, clusters, etc.) that make up the system. Largely, though, without the incorporation of chemically sensitive contrast mechanisms, the identity of the atomic and molecular building blocks remains elusive.

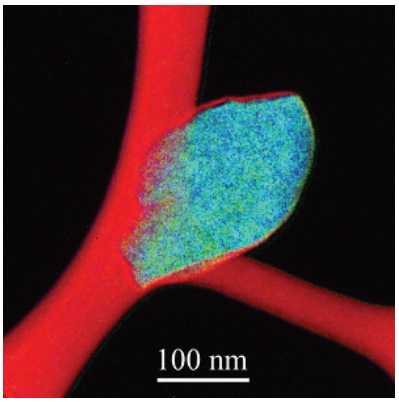

Figure 2.1. Energy-filtered TEM (EFTEM) 3 color elemental map of a manganese oxide nanoparticle showing manganese (blue), oxygen (green), carbon (red) (courtesy of NIST).

The last two decades have witnessed numerous research efforts that sought to provide tools capable of performing measurements on complex, heterogeneous, nanometer-scale systems with the same exacting level of chemical detail that conventional spectroscopic techniques currently provide while ever increasing the level of detail of the object under study. To this end, efforts in proximal probes over the last 20 years have demonstrated that with operation at ultrahigh vacuum and low temperature, spatial resolution down to approximately 0.001 nm can be achieved with STM [6]. With the development of microfabricated cantilevers, AFM can be performed on soft sample surfaces without perturbation [7, 8]. With the advent of intense laser sources and novel methods for generating small optical apertures, chemical contrast mechanisms that rely on vibrational spectroscopy, dielectric spectroscopy and nonlinear spectroscopy have all been demonstrated at length scales well below the diffraction limit of light in near-field microscopies [9-12].

While scanned probes were transitioning from a laboratory curiosity to a reliable and robust measurement tool, an equally impressive revolution was underway in the electron microscopy community [13]. The availability of brighter, coherent field emission electron sources* and the

Technological Advances in Nanocharacterization

- Demonstration of atomic scale chemical sensitivity and mapping.
- Tomography for the morphology and structure determination of very small organic and biostructures as well as inorganic materials and devices.
- Electron holography to image and measure electrical, magnetic, and thickness properties at high resolution.
- Dual-use characterization and fabrication tools such as dual-beam focused ion beam (FIB) and scanned probes allow us to cut, mill, move, glue, and place nanoscale pieces, lines, and even atoms for building and shaping simple nanodevices and artifacts at the same time as we image and characterize them.
- Sample throughput, including data collection and interpretation, has increased dramatically. As an example, TEM has moved from approximately one specimen every few days to, at times, over 10 specimens a day in semiconductor manufacturing facilities.

* Field emission sources were actually first developed in the late 1930s and early 1940s, but made their way slowly into use because of significant technical problems.

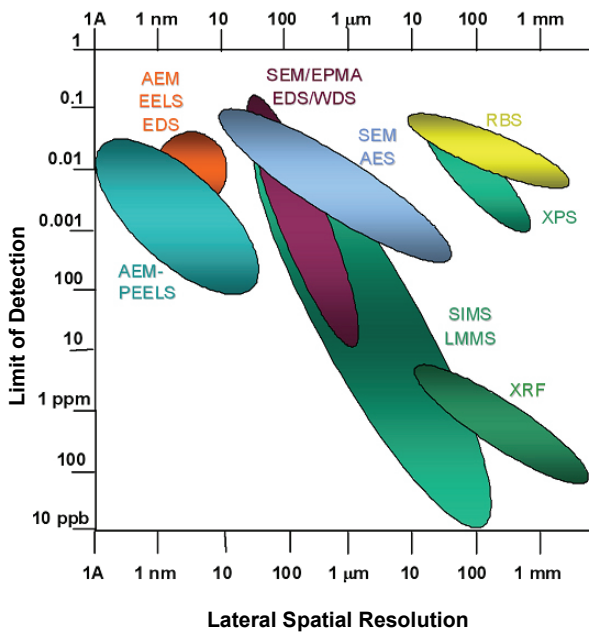

Figure 2.2. Trade-off of trace species detection with spatial resolution (courtesy of NIST).

advent of aberration-corrected lenses [14] enabled sub-0.2 nm spatial resolution imaging with compositional mapping and electron holography [15].

These advances have seen parallel accomplishments in low-voltage scanning and transmission electron microscopy, high-throughput and high-spectral resolution X-ray detectors, and relaxed vacuum requirements through the use of variable pressure instruments enabling the electron microscope to become a tool of tremendous utility to materials characterization. Combining these electron beam capabilities with focused ion beam (FIB) and assisted deposition methods, the dual-beam FIB has become an almost routine machining, manipulation, fabrication, and characterization tool at the nanoscale. The effect of such instrumentation can be seen in almost all fields, from cellular function to magnetic storage media. Many things that are possible and practical today were not possible 20 years ago.

Advanced computer control with digital and spectral imaging approaches has revolutionized all the microscopy methods, allowing quantitative spatially resolved spectroscopy and higher throughput of specimens with easier, more reliable acquisition. Although full automation has not yet been achieved, there is a definite trend in that direction. Another growing trend is remote instrument operation or telepresence for long-distance scientific collaborations [16, 17] to better use unique scientific equipment, especially at Federal laboratories.

The current state of the art might best be viewed as a multidimensional parameter space in which trade-offs are made between spatial resolution and sensitivity, chemical speciation and sampling volume, and speed of data acquisition and detection limits. Figures 2.2–2.4 indicate some of the trade-off issues for different measurands and current metrologies. These figures show approximate ranges of application of various measuring methods based on the best information available.

The current state of the art often reflects a trade-off between one metrology need at the expense of another. To establish the extent of chemical heterogeneity within a sample, one may have to accept something less than a Cartesian coordinate known to ±0.01 nm for each of the atoms that constitute the sample. Similarly, the size and complexity of a structure that can be mapped may reflect a trade-off in time spent on the analysis and the detection limit that is realized.

GOALS, BARRIERS, AND SOLUTIONS

Instruments are needed that are laboratory-based and that push the limits of what can be realized in terms of spatial resolution, chemical sensitivity, speed of data acquisition, and time resolution. At the same time, instrumentation will also be needed that is robust, amenable to production environments, and affordable [18].

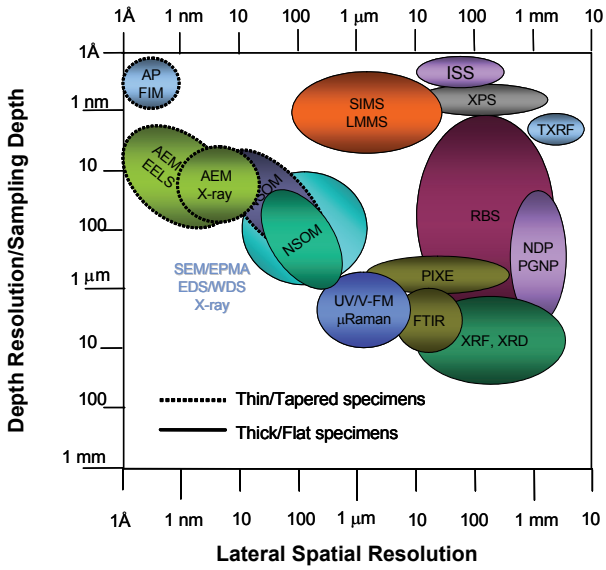

Figure 2.3. Trade-off of depth resolution versus lateral resolution (courtesy of NIST).

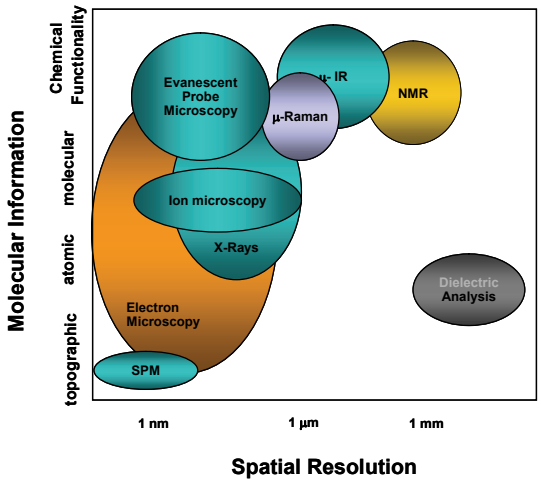

Figure 2.4. Trade-off of chemical specificity with spatial resolution (courtesy of NIST).

For nanoscale characterization of *chemical composition and structure*, a new suite of measurement capabilities will be required. The instrumentation that emerges to meet these needs will require standards and calibrations for the underpinning metrology that cannot be provided by existing metrology. In addition, the processing of data will need to be integrated with the measurement process to a far greater degree than is currently done. Some of this data processing will build on modeling and simulation of the measurement process itself, and some will require merging of data from multiple measurements into a single representation. New instrument development is needed to address improved resolution and sensitivity, increased speed of data acquisition and data reduction, and entirely new or integrated measurement approaches.

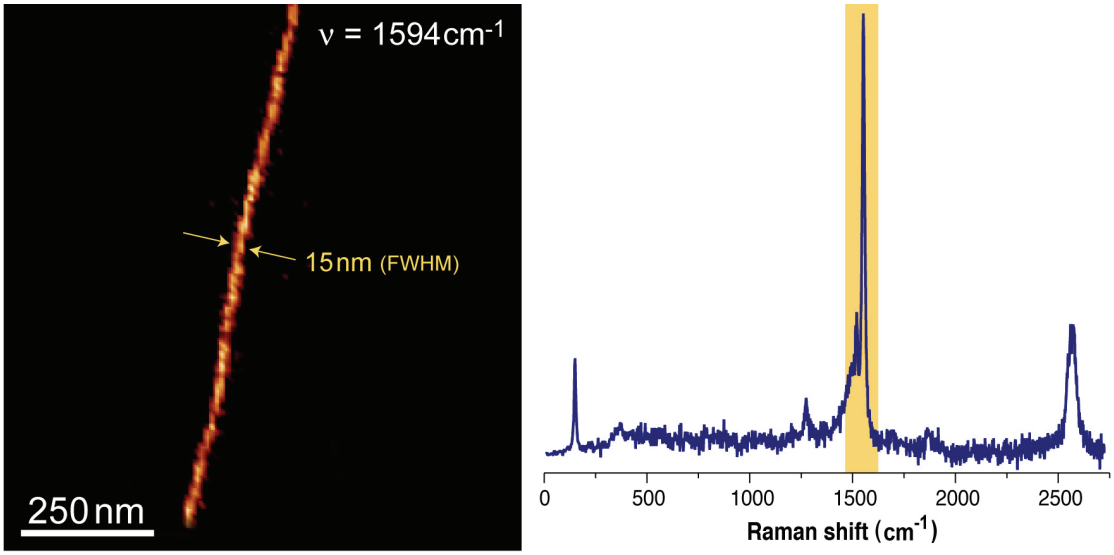

Figure 2.5. Near-field Raman image of a single-walled carbon nanotube (SWNT) and associated Raman scattering spectrum. The contrast in the image reflects the intensity of the G-band (highlighted in the spectrum). The resolution in near-field Raman imaging is determined by the sharpness of a laser-irradiated metal tip, 15 nm in the present case (courtesy of Neil Anderson and Lukas Novotny, The Institute of Optics, University of Rochester).

Although many of the barriers to existing metrology systems and approaches can be overcome through evolutionary advances, other nanoscale characterization needs will require significant breakthroughs which are not expected to occur without a focused effort. In particular, the ability to characterize multiphase systems on the nanoscale will be critical to the assessment of a broad range of systems and will require metrologies that go well beyond what has been developed for periodic, ordered, or uniformly flat materials.

Table 2.1
Key Challenges and Barriers for Nanocharacterization

Metrology	Challenges/Barriers
Chemical Composition	• Multiphase capability • Merging of data from multiple measurements • Standards and calibration • Resolution, sensitivity, and speed
3D Characterization of Structures	• Spatial and spectral resolution and specificity • Data acquisition speed and throughput limitations • Synthesis of 3D information from 2D datasets • Merging of data from different metrology tools • Measurement artifacts
Dispersion of Materials in a Matrix	• Non-existent techniques for examining dispersibility of nanomaterials in a matrix • Poor sensitivity, slow, non-scalable methods
Interface Characterization	• Non-destructive characterization • Buried or embedded interfaces • Interface stability • Probe robustness • Preserving process/sample *in situ*
Speed of Characterization	• Detector speed, efficiency and sensitivity • Source brightness • Software for smart analysis, fast data collection and processing • Fast automated sample preparation
Sample Preparation and Handling	• Inability to extract information about 3D arrangement of atoms • Manipulation of particles • Non-destructive sample sectioning

The ability to examine complex structures in three dimensions is another key barrier that must be addressed if characterization on the nanoscale is going to be capable of addressing the structures that have been envisioned. Measurement strategies that enable data taken using one tool to be merged with complementary data taken from a separate tool will be required to extract full characterization of many of the structures that are anticipated. To make measurements at this dimensional and compositional level, quality control capabilities will need to be developed to ensure that the physics associated with nanostructures do not contribute to artifacts in the measurement system.

Sample preparation remains a pivotal question for both first surface and transmission measurements. The need to characterize the internal structure of complex heterogeneous systems

has driven the metrology community to adopt new techniques that allow one to not disrupt the location of atoms within the sample while exposing the interior of the sample to analysis.

Microtomy, sectioning, depth profiling, and selective erosion with focused ion beams have provided new ways to minimize sample damage while presenting true renderings of interior regions of condensed matter systems. Still, there is a limited ability to peer inside a specimen of interest and to extract information regarding the 3D arrangement of atoms. Issues in sample handling and sample preparation will present new limits on what can be characterized for nanoscale systems, as new methods will be required to manipulate particles, along with refined approaches to sample sectioning that do not affect the nanoscale system.

Other key challenges exist in the *characterization of interfaces* and *dispersion of nanoscale materials* within a matrix. There is a critical need to characterize properties—both chemical and physical—of interfaces at the nanoscale, as these affect the performance of devices and systems. Dispersion of nanoscale materials is also an important indicator of performance and functionality. Dispersibility is of particular importance for nanocomposites, and for any device requiring uniformly distributed nanostructures, such as specialty coatings or sensors, and dielectric films. No techniques are currently available that are capable of providing information about dispersibility within a matrix.

R&D INVESTMENT AND PRIORITY RESEARCH NEEDS

The priority challenges of nanoscale characterization fall along four interrelated components of metrology: (1) the ability to characterize nanoscale structures in three dimensions, (2) the ability to acquire nanoscale data in a timeframe that supports timely interpretation of the results, (3) the ability to measure complex structures with nanoscale compositional heterogeneity, and (4) the ability to establish the dispersion of nanoscale materials. Effort should focus on those techniques that will have the greatest effect on existing needs and industries and will enable new breakthroughs and promote the commercialization of nanotechnology. Development efforts should be conducted with consideration of practical constraints, including safety, reliability, time-to-market, and cost effectiveness.

Along these lines, six priority challenge topics have been developed to achieve goals and overcome barriers to viable nanocharacterization methods. These topics are summarized below and outlined in more detail in Priority Topics 2.1–2.6.

- *3D characterization of individual nanostructures:* characterization of the structure, function, and chemistry of nanostructures. This includes developing a suite of tools and techniques that will allow a detailed characterization of three-dimensionally complex nanostructures at relevant time and length scales.
- *Speed of characterization:* increased speed of characterization to enable productivity improvements, high-throughput and dynamic time-resolved capabilities. The result will be an improved understanding of materials and accelerated commercialization of nanoscale materials.
- *Interface characterization:* characterization of the chemical and physical properties of interfaces (buried, organic–organic/inorganic–organic/inorganic–inorganic) at the nanoscale. Techniques would be nondestructive and include identification of atomic and structural characteristics as well as composition, defects, and anomalies.
- In situ *characterization of interface phenomena:* full understanding of non-equilibrium (reaction) processes at the nanoscale. This would include growth of films and growth of atomic

species into clusters and into particles under controlled atmospheres, temperatures, pressures, fields (electric, magnetic), and other parameters.

- *Quantitative measurement of dispersion of nanoscale materials in a matrix:* development of a robust, quantitative, efficient method to evaluate the dispersion of nanoscale materials in a given matrix, from synthesis all the way to the final manufactured part. Methods should allow measurement of dispersion over all length scales, provide statistically valid results, and be capable of measurements in solution and solid matrices, as well as measuring nanoscale materials suspended in aerosols.
- *Measurement of processes inside of cells:* development of the capability to completely identify and track biological processes at the molecular scale in living cells. Tools should cover mapping protein interactions with high parallelism, imaging cell function in three dimensions, and informatics and protocols for shaping and collecting system biology information.

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

The scientific and technical infrastructure needs for nanocharacterization are addressed through the priority grand challenge topics outlined in the previous section. A combination of instrumentation and metrology tool development, studies in fundamental science and theory, and test and validation will be required to create the needed capabilities for nanocharacterization.

New instrumentation and methods will require supporting standards and calibration. A specific requirement is the development of reference materials with known structure, composition, and function.

IMPLEMENTATION STRATEGIES

Successful implementation will depend on effective collaboration and cost-sharing between various Federal agencies and industry stakeholders. Federal laboratories and universities will have a key role in methods development; all stakeholders should be involved in experimental validation and demonstration.

To move forward, meetings and workshops should be held with potential partners and stakeholders to refine the R&D pathway, milestones, and budgets. Current and proposed funding for this area needs to be evaluated and the potential shortfalls identified. These interactions could fuel the development of a nanotechnology roadmap for nanocharacterization that further defines and guides research.

As technology development progresses, researchers as well as end-users should work with regulatory agencies to understand the risks. Cross-cutting multiagency research programs analogous to the human genome program could be established to accelerate and guide development. These could involve agencies such as the National Institutes of Health (NIH), National Science Foundation (NSF), Department of Commerce, Department of Energy (DOE), and Department of Agriculture.

Priority Topic 2.1. Nanocharacterization Grand Challenge
Three-Dimensional Characterization of Individual Nanostructures

VISION AND GOALS In the future, technology will be available to characterize the structure, function, and chemistry of nanostructures. The supporting goal is to develop a suite of tools and techniques that will allow a detailed characterization of three-dimensionally complex nanostructures at relevant time and length scales.

KEY PERFORMANCE REQUIREMENTS

- Composition analysis with the sensitivity of a few atoms
- Characterization of structural properties (dimension, crystalline structure, heterogeneities) on a scale of 1 nm
- Characterization of the three dimensional (3D) relationship of components and their interfaces
- Ability to measure physical properties such as electrical, magnetic, catalytic, electrochemical, and mechanical properties

COMMERCIAL APPLICATIONS

This is an enabling technology broadly applicable to many industries and user communities:

- Semiconductor device characterization
- Production of catalyst-by-design materials
- Biotechnology
- Environmental and aerosol
- MEMS/NEMS
- Coatings and thin films
- High energy-density materials (HEDMs)

CHALLENGES & BARRIERS

- Inadequate spatial and spectral resolution and chemical sensitivity
- Lack of methods to synthesize 3D information about the sample from 2D datasets
- Data acquisition speed and throughput limitation of existing equipment
- Lack of 3D heterogeneous standard reference samples and calibration/measurement standards

SCOPE OF R&D

- Develop 3D heterogeneous reference samples
- Characterize probes and probe-sample interactions
- Develop better instrument stages
- Develop data processing methods to integrate and reconcile diverse imaging modes and spectroscopies from the same field of view
- Automate existing tools to enable new measurement modes

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing among agencies (e.g., DOD, DOE, NIH, NIST, NASA, NSF, USDA)

Industry – cost-shared partnerships

Federal Laboratories and Universities – research into probe-sample interactions, automation, and reference materials

RISK

LOW — HIGH

Technical Risk

Many complex problems to be solved

Commercial Risk

Moderate risk

IMPACTS

LOW — HIGH

Use across many applications

Reliability of measurements

Relevance to commercial applications

Current technology is non-existent

IMPLEMENTATION STRATEGIES Implementation will be accomplished through partnerships between industry and government that share the development costs associated with high technical risk technologies.

DEVELOPMENT TIMELINE

2005	2010	2015
3D reference samples; probe sample interactions	Integration of diverse imaging modes and spectroscopies from same field of view	Tool automation

Priority Topic 2.2. Nanocharacterization Grand Challenge
Speed of Characterization

VISION AND GOALS Increasing the speed of characterization will lead to productivity improvements, high throughput and dynamic time-resolved capabilities that will improve our understanding of materials and accelerate the commercialization of nanoscale materials.

KEY PERFORMANCE REQUIREMENTS

- Ten fold increase in efficiency and productivity for experiments
- High through-put tools enabling morphological, chemical, structural and property determination for a broad range of nanoscale materials
- Ability to study dynamic processes in near real time (heating and cooling deformation and electrical, mechanical, magnetic and chemical perturbations)

COMMERCIAL APPLICATIONS

Tools will be applicable across multiple manufacturing sectors

- Pharmaceuticals
- High-performance materials
- Semiconductors

CHALLENGES & BARRIERS

- Source brightness improvements for all techniques, better focusing optics needed for ion, X-ray and light sources.
- Order of magnitude improvement in detector speed, efficiency and sensitivity for all techniques (>10X improvements)
- Lack of advanced software for smart analysis (recognition of desired information), fast data collection, processing and analysis
- Lack of fast automated sample preparation techniques for all classes of materials; biological, polymer, ceramic, metal and electronic materials
- Improvements in instrument stability needed.
- Methods for control and perturbation of local sample environment

SCOPE OF R&D

- Select candidate tools in collaboration with vendors and end users, and define development needs for each tool
- Develop standards
- Address development opportunities (detectors, software development, robotics, sample preparation improvements, improved sources)
- Demonstration of tools for selected commercial applications
- Develop standard methods

Partnership Roles and Responsibilities

Industry, Government – cost sharing
Federal Laboratories – Screening and testing of measurement methods; standards and calibrations
Universities – New detector, source and software research
Industry – Experimental validation
Suppliers – Instrument vendors should take the lead in development of new technology
End Users – Participate in validation for selected applications

RISK

LOW — HIGH

Technical Risk

Extended R&D is required for some entirely new tools.

Commercial Risk

Tools must be cost-effective; not increasing speed will slow nano-material development.

IMPACTS

LOW — HIGH

Current technology is non-existent

Reliability and reproducibility of measurements

Use across many applications

Relevance to commercial applications

Flexibility

IMPLEMENTATION STRATEGIES First steps will involve meetings and workshops with potential partners and stakeholders to refine R&D pathway, milestones and budgets. Current and proposed funding for this area and potential shortfalls will be evaluated.

Development Timeline

2005		2010
Select candidate tools and develop standards (2004-2005)	Address development opportunities (2005-2008)	Demonstrate tools, develop standard methods (2009-2010)

Priority Topic 2.3. Nanocharacterization Grand Challenge Interface Characterization

VISION AND GOALS The success or failure of modern devices and systems are determined by a few layers of atoms at interfaces. There is consequently a critical need to characterize the chemical and physical properties of interfaces (buried, organic-organic/inorganic-organic/inorganic-inorganic) at the nanoscale.

KEY PERFORMANCE REQUIREMENTS

- Nanoparticle matrix interactions
- Structural analysis at the atomic or molecular level of any interface
- Compositional bonding and electronic structures at /across/or near the interface
- 3D spatial information at the nanometer scale
- Interface dynamics; temporal chemical and structural changes at the interface (diffusion)
- Transport properties at the interface (carrier mobility, trapped charges; membranes, ion channels)

COMMERCIAL APPLICATIONS

- Semiconductor hybrid systems
- Time released pharmaceuticals
- Antimicrobial food packaging
- High performance composite and coating materials
- Agriculture
- Environmental (mitigation and waste containment)

CHALLENGES & BARRIERS

- Non-destructive characterization
- Buried or embedded interface
- Temporal resolution
- Interface stability (organic-inorganic)
- Separation of information from background or bulk information (S/N)
- Sample preparation

SCOPE OF R&D

- Characterization of inorganic-organic interfaces (replace simulations with real structures/images): film thickness, composition, depth profile of thin film, and at high resolution and high precision
- Two dimensional interface: atomic, structural, and chemical identification of anomalies and defects over microscopic areas; identification of chemical composition between nanoparticles and a matrix
- Non-destructive characterization methodologies

PARTNERSHIP ROLES AND RESPONSIBILITIES

Cost sharing – industry, government (DOD, DOE, NIH, NIST, NASA, NSF, DOA)

Methods development – Federal laboratories, universities, industry

Experimental validation – industry, government, Federal laboratories, universities, suppliers, end-users

RISK

LOW — HIGH

Technical Risk

Physical systems (medium); biological systems (high)

Commercial Risk

Physical systems (low); biological systems (high)

IMPACTS

LOW — HIGH

Organic-inorganic interfaces

Semiconductor device interface

Organic-organic interface

Relevance to commercial applications

Economies and reliability

IMPLEMENTATION STRATEGIES Implementation will be accomplished through technical interactions at meetings and workshops and partnering/collaborating with industry. A key element is to evaluate the proposed and current funding for the area, identify gaps, and plan accordingly. Efforts should include working with regulatory agencies to understand the environmental, health and safety risks. Ultimately, a roadmap should be designed as a tool for future success in developing the needed interface characterization capabilities.

DEVELOPMENT TIMELINE

2005	2010	2015
Characterize inorganic-organic interfaces	Identify atomic, structural, chemical anomalies and defects in 2D interfaces	Non-destructive characterization of interfaces

Priority Topic 2.4. Nanocharacterization Grand Challenge
In situ Characterization of Interface Phenomena

VISION AND GOALS The vision and goal for the future is to fully understand non-equilibrium (reaction) processes at the nanoscale. This would include growth of films, growth of atomic species into clusters, into particles, under controlled atmospheres, temperatures, pressures, fields (electric, magnetic) and other parameters.

KEY PERFORMANCE REQUIREMENTS

- Instrumental techniques using probes with appropriate resolutions and characteristics for the required measurements
- Probe cannot be affected in any substantial way by the material, and probe cannot affect the material during the measurement in any substantial way
- Special environmental cells will be required

COMMERCIAL APPLICATIONS

- Applications will be worth many billions of dollars in economic impacts
- Interfaces in semiconductor materials → control of electronic properties
- Catalysts and catalyst supports and their interactions
- Process measurements for manufacturing of nanoscale pharmaceuticals

CHALLENGES & BARRIERS

- Probe "robustness"
- Preserving sample/process (*in-situ* environment) and integrity (non-intrusive probing)
- Difficulty in developing specialized environmental cells

SCOPE OF R&D

- New methods to characterize growth of nanoparticles in flame synthesis (for making catalysts and other nanoparticulates)
- Specialized cell development for individual probes/techniques
- Real-time imaging methods that can characterize compositional, dimensional and strain changes on the 1 nm to 1000 nm length scale

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing with industry and technology transfer

Industry – cost-shared partnerships with government

Federal Laboratories – screening/testing and technique/ hardware development

Universities – experimental validation

Suppliers – cooperate in R&D with Federal laboratories

End-Users – participate in activities

RISK

LOW — HIGH

Technical Risk

Moderate technical risk for physical systems, higher risk for biological systems

Commercial Risk

Low commercial risk for physical systems, higher risk for biological systems

IMPACTS

LOW — HIGH

Understanding of interfaces

Flexibility

Relevance to commercial applications

Reliability

Economies of use (system-dependent)

DEVELOPMENT TIMELINE

2005	2010	2015
Baseline assessment of existing *in-situ* methods	Assessment of phase-engineered optical fields for interface characterization	Combined *in-situ* dimensional and compositional mapping tools

Priority Topic 2.5. Nanocharacterization Grand Challenge
Quantitative Measurement of Dispersion of Nanoscale Materials in a Matrix

VISION AND GOALS The goal for the future is to develop a robust, quantitative efficient method to evaluate the dispersion of nanoscale materials in a given matrix, from synthesis all the way to the final manufactured part.

KEY PERFORMANCE REQUIREMENTS

- Measurement of dispersion over all length scales
- Statistically valid results
- Fast and simple to use
- Capable of measurements in solution and solid matrices and aerosols in air

COMMERCIAL APPLICATIONS

- Nanocomposites, clarity of films
- Any device that requires a uniformly distributed nanostructure, such as specialty coatings, sensors, EMI
- Shielding, thermal/electrical conductivity
- Nano- and meso-porous dielectric films

CHALLENGES & BARRIERS

- No techniques currently exist that are capable of providing information on the dispersibility of nanomaterials in a matrix
- Current methods are either not statistical, qualitative, slow, not sensitive enough, or not scalable

SCOPE OF R&D

- Collect baseline data for current techniques. Include state of dispersion (macro and nano) with techniques that include direct measurements using optical microscopy, optical absorption, laser-induced fluorescence, optical scattering, neutron scattering and x-ray scattering. Expand these tests toward imaging large area samples. Indirect methods should include electrical and thermal conductivity and mechanical strain propagation.
- Develop surrogate tests (e.g., tensile strength, fractography, conductivity, optical)
- Invent or find a technique to differentiate carbon nanomaterials from carbon matrices (polymers)
- Real time, in-line measurements, non-contact, non-destructive

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost sharing with industry

Federal Laboratories, Universities – measurement methodologies

RISK

LOW — HIGH

Technical Risk

Yield loss, product liability issues

Commercial Risk

Failing to develop the capability for dispersion will impede nearly all product development in nanoscale composites

IMPACTS

LOW — HIGH

Processing

Reliability

Yield

Development of commercial applications

DEVELOPMENT TIMELINE

2005	2010	2015
Baseline for current techniques	Develop surrogate tests; differentiate between carbon nanomaterials and matrices	Real-time inline measurements

Priority Topic 2.6. Nanocharacterization Grand Challenge
Measuring Processes Inside Cells and Tracking Particles in Living Tissue

VISION AND GOALS In the future, it is envisioned that the capability to completely identify and track biological processes at the molecular scale in living cells will be a reality.

KEY PERFORMANCE REQUIREMENTS

- Mapping protein interactions with high parallelism
- Imaging cell function in 3-D
- Informatics tools and protocols for shaping, collecting and presenting system biology information

COMMERCIAL APPLICATIONS

- Medicine
- Agriculture
- Health care products
- Drug production

CHALLENGES & BARRIERS

- Complexity
- Variability among cell types
- Ability to make measurements that preserve cell function and pathway function
- Overwhelming volume of data

SCOPE OF R&D

- Identify and test options for non-interfering tagging and/or other molecular pathway quantification tools
- Choose a cell type and map the complete system biome
- Map all cell types

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry – cost sharing, applications

Federal Laboratories and Universities – develop techniques and technology

Government – cost-sharing (DOD, DOE, NIH, NASA, NIST, NSF, USDA) of infrastructure and research funding

Suppliers – pharmaceutical companies, drugs

Chemical companies – new compounds

End users – mediate adoption based upon safety and efficacy

RISK

LOW — HIGH

Technical Risk

Tools are becoming available but time frame for success is unclear

Commercial Risk

Small, incremental success would yield great returns

IMPACTS

LOW — HIGH

High - Success would lead to a growing revolution in the diagnosis and treatment of disease, including drug design, molecular clinical medicine, and would impact other areas such as precision agriculture

IMPLEMENTATION STRATEGIES A key strategy is to develop cross-cutting programs analogous in type to the human genome program, and involve other agencies such as NIH, NIST, NSF, DOE, and USDA.

DEVELOPMENT TIMELINE

2005	2010	2015
Identify/test options for tagging	Map biome for single cell type	Map all cell types

SUMMARY

Metrology appropriate to nanoscale systems will be critical for the development of nanotechnology, both in terms of the fundamental scientific understanding of those systems and in terms of viable commercial activity. Fields such as electron microscopy, scanned probe microscopy, and optical spectroscopy share common challenges of improved sensitivity, better discrimination, brighter sources, faster data acquisition and analysis, and improved resolution. It is widely accepted that no single technique will be able to provide all of the critical metrology for nanoscale systems, and the ability to develop a breadth of capabilities that allow the broadest spectrum of nanoscale systems to be addressed will be of paramount importance to the ultimate effect of the field.

Ongoing challenges reside in the development of instrumentation built with a level of sophistication sufficient to allow their use by scientists in all fields and in the development of a physical understanding of the factors dictating the response of complex, heterogeneous nanometer-scale systems. Future advances are anticipated to include expansion of the array of material characteristics that can be directly probed on the nanometer scale with particular focus on performance properties, ranging from biomedical activity (drugs) to electronic response (molecular electronics).

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3. INSTRUMENTATION AND METROLOGY FOR NANOMECHANICS

Principal Contributing Authors: Clare Allocca and Douglas Smith

SCOPE

Scientific and technical challenges in nanomechanics cover a spectrum of cross-cutting interdisciplinary research areas and therefore require a wide range of instrumentation and metrology. Scientific challenges in nanomechanics may include, for example, the design of new materials with novel mechanical properties based on tailored nanostructures, or understanding how mechanical properties and responses such as stiffness, hardness, and fracture toughness evolve nonlinearly within arrays of nanosystems or components. Instrumentation for nanomechanics may be required to characterize atoms at interfaces and surfaces in nanoscale materials under applied stresses, or to explore the mechanical behavior of functional nanoscale systems such as biological and biomolecular objects. Advanced metrology may be used to interpret unusual mechanical behavior (strengths approaching theoretical limits, adhesion coefficients reversing with load) at nanometer length scales.

Instrumentation and metrology for nanomechanics focus on both state-of-the-art practice as well as gaps in technology for achieving accurate, repeatable measurements of the mechanical performance of nanostructured materials in devices and systems. Because the list is long in applications and nanomechanics is a broad discipline, we chose to narrow the present discussion to a few important target areas for instrumentation and metrology. *This topic therefore covers the specific technical issues associated with the three most common methods currently used for obtaining mechanical property data at the nanoscale—scanning probe microscopy (SPM), nanoindentation, and nanotribology—but also addresses the issue of future instrumentation needs.* Techniques such as diffraction, small-angle scattering, transmission, and grazing incident reflectometry, along with environmental instrumentation including shear cells, cone and plate rheometers, and applied stress equipment (tension, compression and torsion), are becoming more mainstream, but this topic may be addressed at a future workshop.

- Nanomechanical metrology addresses factors critical to the accurate and precise execution and interpretation of measurements. These factors include highly localized variations in defect arrangements, dimensional scaling of properties from continuum (macroscopic) levels to nanometer levels, temporally-varying behavior, and a complex, composite-like behavior often seen in nanomaterial systems.
- The instrumentation required for successful nanomechanics metrology must provide high spatial resolution and an understanding of the contact mechanics associated with tips for SPM, indentation and tribology. Supporting technical areas include high data collection rates, time-resolved measurements, and best practices for testing, tip characterization, and specimen preparation.

Vision for Nanomechanics

In order to be widely used, future nanodevices will require nanomechanical measurements that are rapid, accurate, predictive, well-understood, and representative of a device or system's environment in real time.

VISION

Achieving the vision for nanomechanics will require:

- Global standards that include primary calibration, use of calibration artifacts, standard test methods, and standardized data analysis methods
- Test specimens, platforms and testing techniques that enable measurement of mechanical properties in real application environments and at appropriate length scales
- Models that enable a quantitative connection between mechanical measurements at the nanoscale and relevant material properties
- Multiprobe instrumentation that provides detailed mechanistic information during and after nanomechanical property measurements for proper data interpretation; for example, microstructural changes and chemical changes as a result of the mechanical probing need to be monitored and understood to avoid misinterpretation of results
- Automated metrology platforms that enable integrated, multifunctional, high-spatial resolution, rapid nanomechanical measurements and analysis

Table 3.1
Current State of the Art of Nanomechanical Instrumentation

Instrumentation	Accuracy	Sensitivity	Resolution	Precision	Compatibility with Different Material Systems
Scanning Probe Microscopes	• Greatest challenge • Calibration-dependent • Tip and surface damage contributes to poor accuracy	• Piconewton to nanonewton range • Can be enhanced through chemical modification of the tip	• Subnanometer • Dependent on scanning mode and sharpness of tip	• Dependent on cantilever, environment, and scanner performance	• Limited to materials with $E > 10^9$ Pa
Instrumented Indentation Testing	• Large uncertainties at small forces and displacements • Model-dependent results	• Nanonewton to millinewton range • Can be enhanced by harmonic oscillation	• Large tips not conducive to nanoscale measurements	• Highly dependent on tip shape knowledge	• Limited to materials with $E > 10^6$ Pa
Tribometers and Nanoscratch Testers	• Real area of contact determination is the major limitation • Higher requirements in rigidity because lateral resistance can be large • Force sensors are more sensitive than instrumented indentation testing but less than scanning probe microscopes	• Nanonewton to millinewton range • Vibration and environmental factors affect results significantly • Large variety of tips and cantilever designs	• Depends on tip sizes • Most tips are suitable for microscale measurements • Advanced diamond tips can get below 50 nm radius	• Depth of penetration during sliding can be controlled only to several nanometers • Dependent on environmental control and vibration isolation	• Limited by surface roughness at nanometer scale • Materials limited by tip hardness and system stiffness

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

The measurement of mechanical properties at the nanoscale is currently based on SPM and nanoindentation technologies, in which probes of various sizes and shapes are used to make direct physical contact with a sample surface. A summary of the current state of the art in nanomechanical instrumentation is presented in Table 3.1. Although SPM techniques have been developed to pull polymeric molecules in tension, the majority of nanoscale mechanical testing has been performed by pushing the probe into the sample and using appropriate contact mechanics to evaluate the material response to indentation. Additionally, lateral motion can be applied to measure tribological response at the nanoscale.

Commercial SPM systems have been developed primarily to provide unmatched microscale to nanoscale imaging capabilities. However, their ability to measure mechanical properties at these scales is largely qualitative. This is due to poorly known spring constants, non-ideal (and often unknown) probe geometries that can change significantly with use, nonlinear performance of the piezoelectric scanners, probe instabilities, and lateral motion of the cantilever probe. Modified or custom-made SPM systems have significantly improved mechanical property measurements by using electrostatic or magnetic force feedback, custom-made probe tips, or external measurement and control of scanner motion, but these modifications often limit the imaging capabilities of these noncommercial systems. Limitations in nanoscale imaging capabilities also plague nanoindentation systems, as these instruments have been developed solely to provide quantitative characterization of mechanical properties and behavior. Even the most sensitive of these systems, however, has limited capacity for nanoscale measurements, as the probe geometries currently manufactured have dimensions that, when combined with current signal-to-noise levels, are not appropriate for atomic-scale testing. Accuracy, sensitivity, resolution, precision, and compatibility with different material systems were identified as the important parameters for characterizing the state of this technology.

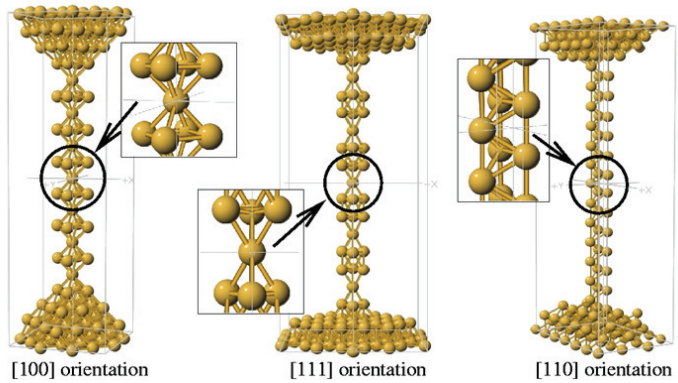

Figure 3.1. Direct experimental measurements of atomic bonding forces often involve consideration of different possible crystalline structures at contacts, as illustrated by these three configurations of a gold nanowire (courtesy of NIST).

GOALS, BARRIERS, AND SOLUTIONS

There are many technical challenges that must be addressed to successfully develop the nanomechanical instrumentation and metrology needed to support the future nanotechnology industry (see Table 3.2). These challenges involve the development of standards and calibration methods, accurate predictive modeling tools, and reliable, fast, multifunctional, quantitative instrumentation. A key approach will be to develop those methodologies with the greatest potential to achieve breakthroughs and enable successful commercialization of nanotechnology.

In the area of *standards and calibration*, consensus standards based on best practice must be developed and adopted by the international community. The challenge is to create standards that

are reliable, robust and machine independent. Traceable force and displacement calibration must be available with subnanonewton and subnanometer resolution, respectively.

Table 3.2
Key Challenges and Barriers for Nanomechanics

Metrology	Challenges/Barriers
Standards and Calibration	• Traceable force and displacement calibration • International collaboration to establish common standards • Methodologies based on reliable data and models • Machine-independent standards • Understanding of nanometer scale surface forces and contact mechanics • Wide experimental dynamic range
Nano-mechanical Modeling of Experiments	• Modeling—computational power, intelligent data storage and mining, length of model development time, capturing physics • Experiment—ability to fabricate and characterize testing fixtures, manufacture and characterize samples; accuracy and traceability for experiments; ability to position/manipulate samples
Integration of Multiple Techniques	• *In situ* probes for imaging, manipulation, chemical bonding and orientation detection at atomic/molecular resolution • Spatial resolution when focusing on a single event • Integration of software, input/output compatibility, control languages • Synchronization of time and position information
High-Throughput Automated Measurements	• Sample preparation—speed, automation, yield, quality, size, conditioning, and material specific issues (polymers, metals, ceramics, glasses, electronics) • Calibration—robust probes, periodic reference specimens or characterizations • Analysis/testing schemes—lack of wide range of testing environments (temperature, frequency); lack of models to describe complex nanoscale mechanical behavior; lack of high-speed methodologies; lack of well-characterized nanoscale probes
Instrument Development	• Tip wear, control, cm to nm positioning • Decoupled lateral and vertical force sensors • Lack of lateral or vertical force calibration standards • Multiple operating mechanisms and environments required for mechanical property measurements • Non-linearity of actuators/sensors • Thermal drift • Quantitative mechanical property mapping is typically a slow, point-by-point process
Measurement Under Real Application Conditions	• Real area of contact • Surface treatments • Robustness • Application-compatible materials • Real-time measurement capabilities • Sub-element specific testing (e.g., interfaces)

The most immediate challenges to be addressed in this area are those related to the calibration and performance verification of existing commercial instruments such as nanoindenters, atomic force microscopes and nanotribology equipment. These instruments require rapid development of internationally-accepted traceable calibration procedures, test procedures, and standard reference

materials for routine verification of machine performance. In the long term, an important standardization and calibration goal is to develop methods for producing highly precise, well-characterized probe tip geometries.

Predictive modeling tools are needed to provide support for product development and manufacturing. Key challenges are adequate computational power, the capability for intelligent data storage and mining, the time and expense of model development, and correctly capturing the physics in the models, especially for atomistic and mesoscale models. Models must be supported by experimental validation. Key challenges in that area include the ability to fabricate and characterize testing fixtures (tips, grips, etc.) and the ability to manufacture and characterize samples. Other issues involve accuracy and traceability and the ability to position and manipulate samples effectively.

The *integration of multifunctional techniques* will help to fulfill the promise of nanotechnology. The primary requirement is the ability to probe nanoscale deformation, image it, and understand the physical and chemical processes occurring in real time. Individually, current instruments such as nanoindentation equipment, atomic force microscopes, and the surface force apparatus can provide only partial information about nanoscale deformation. As a result, obtaining a complete understanding of any specific nanodeformation event is extremely difficult. This is a major barrier for nanotechnology and one that can only be overcome by integrated, multifunctional measurements. Specific technical challenges include the use of various probes focusing with high spatial resolution on a single location, and spatial and temporal synchronization of information gathered by various probes. A key issue will be integration of various probes in terms of software, input/output compatibility, and control languages.

Initial efforts should focus on integrating SPM with one or more spectroscopic capabilities to yield additional useful information. Miniature scanning electron microscopy and X-ray probes should be developed and incorporated into existing and emerging instrumentation. System integration should be undertaken in partnership with equipment vendors and university inventors to provide user-friendly interfaces.

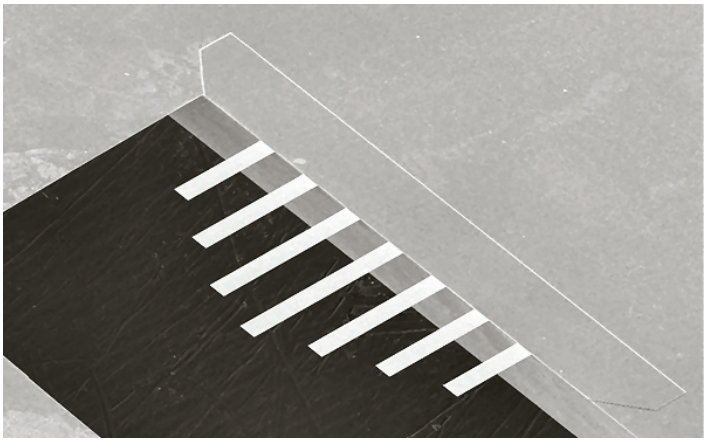

Figure 3.2. NIST is developing a set of standard reference cantilevers with well-defined spring constants for use in the traceable calibration of force for scanning probe microscope systems (courtesy of NIST).

Sample preparation continues to limit nanomechanical metrology. There is a lack of high-speed sample preparation equipment, especially for interrogating the internal structure of nanomaterials. Fast, automated sample preparation techniques are needed for all classes of materials (e.g., biological, polymer, ceramic, metal and electronic), along with a wide range of testing conditions (temperature, environment).

To meet these challenges, near-term efforts should focus on identification of candidate approaches that involve materials suppliers, end users, and instrument vendors, so that the platform is effective and has a ready path to commercialization. Over the midterm, development of new metrology will require joint efforts

from vendors (construction and commercialization), industry (applications and platform testing), government agencies (precompetitive funding opportunities), Federal labs (e.g., NIST for fundamental measurement science), and universities (modeling). This development is multidisciplinary in nature, and the end result should be a commercial platform for high-speed nanomechanical measurements. Longer-term development should focus on expanding platforms to include high-throughput nanoscale testing.

Instrument development is needed to address a number of nanomechanical metrology challenges impeding future progress in nanotechnology. For example, probe tip shape is seldom known and changes during experiments, lateral and vertical force sensors are often coupled, and force calibration methods and traceable standards are not currently available. A key issue is that mechanical property measurements require multiple experimental mechanisms and environments—a capability that does not currently exist.

Independent lateral and vertical force sensors, enhanced positioning accuracy, and greater speed will improve multiple scanning probe methodologies and should therefore be developed in the near term, with continued improvements over time. Calibration standards and methods for lateral and vertical forces are reasonable midterm goals that will require significant collaboration and support from industry, academic researchers, and government labs and policymakers. Tip-shape control and wear minimization are important long-term goals, as they will require significant research and development.

The ability to make *in situ* nanomechanical measurements in real applications will be a requirement for the future success of nanotechnology. Key challenges include being able to make measurements of the real area of contact and the availability of application-compatible probe materials. Tools will need to be robust and have real-time measurement capabilities. A key capability will be subelement-specific testing (e.g., interface, buried layer, quantum dot).

R&D INVESTMENT AND PRIORITY RESEARCH NEEDS

Six priority challenge topics have been developed to overcome the technical barriers and achieve the vision and goals for nanomechanical instrumentation and metrology. These challenge topics are summarized below in order of priority and are described in more detail in Priority Topics 3.1–3.6.

- *Standards and calibration:* addressing calibration and standards needs for mechanical testing at the nanoscale is critical for obtaining the accurate, quantitative material properties necessary for device design and product specifications. Science-based standards must be developed that include traceable primary calibrations, the use of artifact standards, and standard test and analysis methods. This methodology will enable better understanding of mechanical properties at the nanoscale and will facilitate the direct comparison of data from different laboratories and different instruments—an essential step toward international acceptance of test methods and results.
- *Nanomechanical modeling of experiments:* simulation tools are needed to describe the quantitative connection between mechanical measurements at the nanoscale and related material properties. The goal is to have integrated tools that will allow visualization, positioning, sample manipulation, and chemical characterization.
- *Integration of multiple techniques in nanomechanics:* in nanomechanics, the key issues are force measurement sensitivity, knowledge and control of contact area, imaging capability and imaging rate. Current SPM techniques are suitable for imaging nanoscale events but inadequate for measuring nanomechanical properties or efficient manipulation of atoms, molecules, or

nanoparticles. As a consequence, there is a real need for new instrumentation and improved sensors and imaging tools in nanomechanics that integrate various measurement functions.

- *High-throughput automated nanomechanical measurements:* high-speed, quantitative nanomechanical measurements are critical to ensure U.S. dominance in the development of nanomaterials by design. As a result of global competition, the design of functional materials must increasingly rely on lower-cost R&D through combinatorial approaches, incorporation of nanoscale fillers into conventional materials, and architecture control to achieve desired properties at the lowest price. To accelerate this development, new metrology tools will be required to deliver high-speed, quantitative nanomechanical testing. This will include the need for mechanical metrology below 100 nm, obtained in both localized and imaging modes. Current technologies are either too slow (nanoindentation) or not sufficiently quantitative (AFM).
- *Instrument development for nanomechanics:* measurement capabilities need to be improved in many areas, including enhanced positioning capability, increased throughput and axially independent force sensors. Multiple mechanical measurements in a variety of experimental environments should be available with the same nanoscale spatial resolution and data acquisition times as standard topographic scanning. These improvements would allow mechanical properties to be quantitatively mapped at the nanoscale, with obvious benefits to nanoscale engineering.
- *Experimentation/testing under real application conditions:* the goal is to successfully develop test specimens, platforms, and testing techniques that will enable testing at appropriate length scales in real application environments.

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

Instrumentation and metrology for nanomechanics rely on a strong spectrum of state-of-the-art instrumentation and facilities to accelerate research and development. The NNI is investing in a strong and evolving national infrastructure that supports new instrumentation for nanomechanics through centers of excellence, national facilities, user networking programs, and complementary computational centers for nanoscience and technology. Such an investment of multifaceted, governmentally-supported networks of centers and research facilities is essential for advancing new discoveries, taking innovations to commercialization, and increasing educational resources. For example, although the NSF is supporting the National Nanotechnology Infrastructure Network and Centers of Excellence through a university program, the Department of Energy is supporting the construction and operation of five national Nanoscale Science Research Centers with open, free access to academia, government and industry researchers. NIST has built a new Advanced Measurement Laboratory with nanofabrication facilities and high-accuracy measurement laboratories. Through research done in these facilities, innovations and applications based on fundamental science and metrology at the nanoscale in nanomechanics will directly benefit the Nation's economic growth.

Priority Topic 3.1. Nanomechanics Grand Challenge
Standards and Calibration

VISION AND GOALS It is envisioned that science-based standards with international acceptance will be developed. These will include primary calibration, artifact standards, and standard methods for testing and analyzing data for establishing the quantitative measurement of mechanical properties at the nanometer scale.

KEY PERFORMANCE REQUIREMENTS

- Nanometer – scale contact
- Wide dynamic range, covering many decades in force, displacement and frequency
- Validated and commonly accepted set of standard test methods for measurement of basic mechanical properties at the nanoscale

COMMERCIAL APPLICATIONS

- Thin films for electronics, MEMS, and other devices
- Bio-interactions
- Material development
- Product specifications

CHALLENGES & BARRIERS

- Traceable force and displacement calibration
- Collaboration in the international community to establish common standards with coordinated funding streams
- Establishment of methodologies based on reliable data and models
- Machine-independent standards
- An understanding of surface forces and contact mechanics at the nanometer scale
- Wide experimental dynamic range (many orders of magnitude in force, displacement and frequency)

SCOPE OF R&D

- Traceable force and displacement calibration procedures for commercial nanoindenters, atomic force microscopes and other nanomechanical test equipment
- SRM's/CRM's for Young's Modulus of bulk ceramic and metallic materials
- Standard methods for tip shape calibration
- Internationally accepted guidance documents for measurement protocols with standardized reporting procedures
- Methods for fabricating arbitrary tip shapes to high accuracy (tight tolerances)

PARTNERSHIP ROLES AND RESPONSIBILITIES

- **Industry** – application/verification of standard methods and calibration procedures
- **Government** – coordination/funding
- **Federal Laboratories** – development of state-of-the-art instrumentation, calibration and test methods; basic metrology infrastructure
- **Universities** – fundamental R&D on experimental techniques, modeling/ interpretation

RISK

LOW — HIGH

Technical Risk

Verifying force/displacement data

Commercial Risk

Industrial/international consensus

General Risk

Significant resource requirements

IMPACTS

LOW — HIGH

Product standards

Improved quality of scientific results

Regulatory standards

IMPLEMENTATION STRATEGIES Implementation will be accomplished through national and international workshops and round robins, and development of a roadmap for standards.

DEVELOPMENT TIMELINE

2005	2010	2015
SRMs/CRMs for bulk ceramics and metallics; traceable force/displacement calibration (2005-2006)	Nanoscale tip characterization (2007)	Internationally accepted guidance for measurement protocols (2010)	Highly precise tip shape manufacturing

Priority Topic 3.2. Nanomechanics Grand Challenge
Nanomechanical Modeling of Experiments

VISION AND GOALS Simulation tools are needed to describe the quantitative connection between mechanical measurements at the nanoscale and related material properties. The vision is to have integrated tools that will allow visualization, positioning, sample manipulation, and chemical characterization.

KEY CAPABILITIES

- Close coordination among theory, computation, and experiments
- Multi-scale modeling: Atomistic to mesoscale to continuum
- Modeling time scales compatible with experiment
- Accurate, high precision, controlled, and traceable experiments

COMMERCIAL APPLICATIONS

- Semiconductor manufacturing (information technology)
- Data storage
- NEMS/MEMS
- Sensors/Actuators
- Biomedical (diagnostic and therapeutic)
- Commercial/protective coatings
- Nanocomposite/nanoparticle applications
- Nanofabrication (stamping, imprinting)

CHALLENGES & BARRIERS

Modeling

- Computational power
- Intelligent data storage and mining
- Length of time to develop models
- Capturing physics in the model (especially for atomistic and mesoscale)

Experiment

- Ability to fabricate and characterize testing fixtures (tips, grips, etc.)
- Ability to manufacture and characterize samples
- Accuracy and traceability for all experiments
- Ability to position and manipulate sample

SCOPE OF R&D

Experimental

- Standards and calibrations
- Integrated tools to allow visualization, positioning, sample manipulation, and chemical characterization
- Modeling input to design experiments

Modeling

- Experimentally/ab initio validated interatomic force fields
- Coupling between length, time and temperature scales
- Models that accurately represent experiments

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry, Government, Universities – all need to participate

End Users – validation

Suppliers – make technology accessible and participate in standardization

RISK

LOW — HIGH

Technical Risk

Some new tools and integration are needed

Commercial Risk

Translation to commercial use difficult for some applications

IMPACTS

LOW — HIGH

Current technology non-existent

Reliability, reproducibility of measurements

Use across many applications

Flexibility

Economics of Use

IMPLEMENTATION STRATEGIES Key strategies for implementation include focused workshops to gain consensus on path forward, funding, partners and other issues; promoting funding initiatives to study fundamental and new perspectives; and innovative partnerships with industry.

DEVELOPMENT TIMELINE

2005	2010	2015
Standards and calibration; validated interatomic force fields	Integrated tools; coupling between length and time scales	Models that represent experiments

Priority Topic 3.3. Nanomechanics Grand Challenge
Integration of Multiple Techniques

VISION AND GOALS The long-term vision is to achieve multifunctional, integrative nanomechanical instruments with *in situ* capability.

KEY PERFORMANCE REQUIREMENTS

- *In situ* probes (controlled environment, i.e., temperature, pressure, imaging medium) with simultaneous, spatial, mechanical and chemical information at atomic/molecular resolution
- High bandwidth measurement capability

COMMERCIAL APPLICATIONS

- Biomedical devices
- MEMS/NEMS, semiconductors, data storage
- Sensors and actuators
- Aerospace
- Nanomaterials testing

CHALLENGES & BARRIERS

- *In situ* probes for imaging, manipulation, chemical bonding and orientation detection at atomic/molecular resolution
- Spatial resolution of probes focusing on a single event location
- Integration of probes – software, input/output compatibility, control languages
- Synchronization of information gathered on time and position

SCOPE OF R&D

- Miniature SEM probes, simultaneous multi-probe heads for imaging, manipulation of nanomechanical events
- Integration of nanoindenters with TEM for direct dislocation imaging under known stresses and displacements
- Integration of SPM with one or more spectroscopic capabilities (e.g., fluorescence, Ramen, x-ray spectroscopes)
- Chemical sensing probes with molecular spatial resolution, sensitivity to detect changes in molecular configuration, geometric shape, atomic lattice
- Full integration of simultaneous mechanical, compositional, and transport property measurements

Partnership Roles and Responsibilities

Government – funding based on peer-review; promotes public awareness of economic benefits and quality of life.

Federal Laboratories – demonstrate prototypes; integrate university R&D; form partnerships with universities, industry.

Industry – Leads commercialization of instrumentation; defines information required for commercialization of nanotechnology; supports university research.

Universities – pioneer new concepts; develop prototypes.

RISK

LOW — HIGH

Technical Risk

Physics are not well understood, needs far exceed current state-of-the-art manufacturing capabilities.

Commercial Risk

Significant international competition.

IMPACTS

LOW — HIGH

Technology will provide key information to enable successful development of nanoscience and nanotechnology.

IMPLEMENTATION STRATEGIES Successful implementation will require enhanced, leveraged instrument-driven funding. Funding initiatives should be created to develop the basic understanding necessary to demonstrate multi-technique integration. A key strategy will be to establish coordinated meetings and workshops for refining and assessing R&D targets and milestone and accomplishments.

Development Timeline

2005	2006	2008
Define critical information requirements; develop new probes suitable for integration (small, compatible with existing probes)	Partnerships with equipment vendors to initiative integration; first prototype demonstration (2007)	Multiprobe instrumentation commercially available (2008)

Priority Topic 3.4. Nanomechanics Grand Challenge
High-Throughput Automated Nanomechanical Measurements

VISION AND GOALS An automated metrology platform that enables integrated, rapid nanomechanical measurements and analysis is envisioned. This high throughput platform would serve to accelerate new materials development and manufacturing quality controls. The goals are a ten-fold reduction in R&D cycle time for mechanical testing, and quantitative mechanical properties measurement on length scales <100 nm with precision <5 percent.

KEY CAPABILITIES

- Sample preparation
- Data analysis
- Sample indexing
- Reporting and feedback mechanisms
- Testing and acquisition
- Database creation and archiving
- On-line calibration

COMMERCIAL APPLICATIONS

- New material development
- New formulation development
- Customization capability
- Engineered surfaces
- Quantified quality testing (warranty)

CHALLENGES & BARRIERS

- **Sample preparation** (solution, solids, films, parts – metallography, microforming, spin-coating, control of printing, ink-jet). Issues include speed, automation, yield, quality, size, conditioning, and material specific issues (polymers, metals, ceramics, glasses, electronics)
- **Calibration** – robust probes, periodic reference specimens or characterizations, suite of materials
- **Analysis/testing schemes** – lack of wide range of testing environments (temperature, frequency); lack of models to describe complex nanoscale mechanical behavior; lack of high speed methodologies for mechanical testing; lack of well-characterized nanoscale probes

SCOPE OF R&D

- Sample preparation - evaluation of more rapid testing schemes, flexible integrated sample preparation, combinatorial libraries
- Mechanical property mapping at high frame rates, high speed local testing
- Integrated modeling and simulation for many material types to convert instrument output to properties
- Wide temperature and frequency capability
- Flexible, "smart" testing algorithms that can suggest testing protocols (time - dependent responses)

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry – industry consortia, instrument manufacturers

Government – financial support, sample preparation, data management

Federal Laboratories – facilitate consortia; method validation and data management

Universities – fundamental science (e.g., sample preparation, informatics)

RISK

LOW — HIGH

Technical Risk

High technical and commercial risk for instrument manufacturers and industries

Commercial Risk

IMPACTS

LOW — HIGH

Sample preparation

Accelerated materials development

Leveraged to variety of materials

Competitive advantage in materials lifetime

IMPLEMENTATION STRATEGIES Key strategies for implementation will include 1) development of materials roadmaps by key industry segments; 2) creating an education and training infrastructure to support nanotechnology; 3) ensuring that funding programs are attractive to industry and vendors (funds available, protection of intellectual property); 4) incorporating modularization to avoid duplication; and 5) making expertise available, or creating open access measurement technology centers.

DEVELOPMENT TIMELINE

2005	2006	2009
Candidate tools selection with vendors, end-users, manufacturers	Sample prep, high speed acquisition, mechanical probes, evaluation of models, codes, informatics, process/feedback control, combinatorial methods (2005-2008)	Prototype testing

Priority Topic 3.5. Nanomechanics Grand Challenge
Instrument Development for Nanomechanics

VISION AND GOALS A high throughput, high spatial resolution reliable quantitative measurement technology is envisioned, encompassing a suite of multiple mechanical measurements in nanometer scales.

KEY CAPABILITIES

- Throughput (20-50 wafer/hour, video rate scanning, producing dimensional property data)
- Modules with a few percent accuracy (long term)
- Properties capabilities: viscoelasticity, sub Hz to GHz, adhesion, pulling (tensile)
- Drift-free platform, non-degradable tip
- Non-probe-based nanoscale *in vitro* measurements
- *In situ* measurements (fluid)
- Variable temperature measurements
- Mechanical characterization system

COMMERCIAL APPLICATIONS

- Semi-conductor/device industry
- Nanomaterials development
- Life sciences (medical, environmental)

CHALLENGES & BARRIERS

- Tip wear, control, cm to nm positioning
- Lateral and vertical force sensors are often coupled
- Lack of lateral or vertical force calibration standards
- Multiple operating mechanisms and environments are required for mechanical property measurements
- Non-linearity of actuators/sensors
- Thermal drift
- Quantitative mechanical property mapping is typically a slow, point by point process

SCOPE OF R&D

- Low wear, controlled tip shapes; high resolution, accurate, stable and rapid tip positioning and scanning
- Mechanically robust, chemically and biologically tailorable tips
- Sub-nanometer positioning accuracy (lateral and vertical)
- Calibration standards for lateral and vertical forces
- Decoupled force sensors in lateral and vertical directions
- Multiple measurement capabilities (static, viscoelastic, ultrasonic) and multiple environment capabilities (fluid, vacuum, variable temperature)

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – fund R&D
Industry – accuracy, speed, decoupled sensors, multiple capabilities, tip shape control, force calibration and other standards
Federal Laboratories – decoupled sensors, force calibration standards, multiple capabilities
Universities – tip shape control, multiple capabilities, speed

RISK

LOW — HIGH

Technical Risk

Standards require government, industrial, and academic collaboration and support

Commercial Risk

Cost versus performance trade-off

IMPACTS

LOW — HIGH

Standards development

Reliable quantitative results

Accurate measurements from specific positions/ materials/ conditions

IMPLEMENTATION STRATEGIES Key strategies for implementation include focused workshops to gain consensus on path forward, funding, partners and other issues; the promotion of funding initiatives to study fundamental and new perspectives; and innovative partnerships with industry.

DEVELOPMENT TIMELINE

2005	2010		2015
Independent lateral and vertical force sensors	High accuracy positioning; improved scan and positioning	Low cost lateral and vertical force standards and methods	Control over tip geometry and wear

Priority Topic 3.6. Nanomechanics Grand Challenge
Measurement Under Real Application Conditions

VISION AND GOALS The goal for the future is to successfully develop test specimens, platforms, and testing techniques that enable testing under real application environments and at appropriate length scales.

KEY CAPABILITIES

- Sensitivity to actual contact force
- Non-ambient pressure/temperature/humidity
- High frequency/friction
- Appropriate length scales and realistic material combinations
- Degradation detection (e.g., fatigue/fracture/corrosion)

COMMERCIAL APPLICATIONS

- *In vivo* bio-applications (invasive)
- Biomedical devices (drug delivery, lab-on-chip)
- Gas/chemical/bio-sensors
- Information storage, optical storage
- Extreme-environment sensors
- High-g accelerometers
- RF micro-switches
- Shape-memory materials for high-density information (read/write)
- NEMS/MEMS, multi-array production

CHALLENGES & BARRIERS

- Real area of contact
- Surface treatments
- Robustness
- Application-compatible materials
- Real-time measurement capabilities
- Sub-element specific testing (e.g., interface, buried layer, quantum dot)

SCOPE OF R&D

- MEMS, hard disk drives, multi-array platforms
- Extreme-temperature/harsh environment MEMS (e.g., SiC), molecular manipulators
- Shape-memory/quantum storage information

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry – fund R&D, establish standards

Federal Laboratories – measurements, standards development

Universities – provide tools, detection

RISK

LOW — HIGH

Technical Risk

Some existing tools are readily available; integration is needed but appears feasible; extreme environments are high risk

Commercial Risk

MEMS/NEMS exist or are close to reality; guardian/shape memory/ molecular manipulators need more R&D, e.g., advances in biomaterials testing, sensors

IMPACTS

LOW — HIGH

Use across many applications

Enabling measurement tools for key emerging nanotechnologies

Relevant to several known applications (e.g., biomaterials testing and sensors)

IMPLEMENTATION STRATEGIES Key strategies for implementation will include 1) co-location of workshops with main society meetings, 2) more focused workshops, 3) evaluation of funding opportunities and coordination of activities whenever possible, and 4) possible round robins.

DEVELOPMENT TIMELINE

2005	2008	2012
MEMS, hard disk drives, multi-array platforms	Harsh environment MEMS, molecular manipulators	Shape-memory/ quantum storage

Concerted improvements in tip geometries and tip materials will be essential for advancing the spectrum of nanomechanics, systems, and devices. Advancement in time-resolved equipment and *in situ* environments will be required to support increased innovation and testing. Significant improvement in parallel computing and interactive software, real-time visualization, and data manipulation will also be essential for the success of a national nanomechanics program. Training and workforce development follow hand-in-hand, where education and scientific researcher resources improve the economy, national competitiveness, and creative advantage, so as to take nanomechanics from science and technology to commercialization.

IMPLEMENTATION STRATEGIES

Implementation will depend on the collaborative activities and support of government, industry, national laboratories, and universities. A key strategy will be to hold workshops (domestic, international) that further refine and identify the path of research and commercialization efforts. An important component is the evaluation of funding activities and shortfalls, potential leveraging of funds, and the establishment of funding programs that are attractive to vendors and industry in terms of dollars as well as other issues (e.g., intellectual property protection). Another approach is to leverage state/regional initiatives to promote local nanotechnology expertise and equipment as well as open technology centers at universities. These could operate in parallel with joint programs, using coordinated inter-regional funding. Leveraging of the expertise at the government laboratories is essential. The development of technology-specific roadmaps is another approach that could further define future activities.

SUMMARY

Future nanodevices will require nanomechanical measurements that are rapid, accurate, predictive, and well understood. These measurements must be able to reproduce a device or system's environment in real time. Realization of this vision requires the successful integration of two distinct scientific disciplines, each challenging in its own right. First is the experimental ability to physically deform nanoscale volumes of material using instruments that accurately determine both the stress applied to the material and the resulting strain. Second is the theoretical and modeling capability to understand and predict the mechanical behavior of matter at the atomic level. If these two capabilities can be developed simultaneously and interactively, a robust and extremely powerful understanding of atomic-scale deformation will evolve that will enable not only detailed analyses of the mechanical performance and reliability of existing materials and devices, but also the accurate prediction of the properties of devices and materials that do not yet exist.

The current state of the art in both experiment and theory falls far short of the capability required to realize this vision. Existing instruments for measuring mechanical properties at the nanoscale typically apply poorly calibrated forces to a specimen through poorly characterized physical contacts, producing complex, three-dimensional stress–strain fields that are difficult to analyze. Modeling the mechanics of a very small system of atoms (up to several hundred) can be done accurately from first-principles calculations, but better atomic potentials must be developed for larger systems, and methods must be developed for the interfacing of models that operate on different length and time-scales. These barriers can be overcome through improvements in both experimental and modeling accuracy, driven by an iterative process of mutual validation. That is, accurate traceable experimental data can be used to develop better atomic potentials, and models using improved potentials can in turn enable a more accurate interpretation of experimental data. In addition to improved accuracy in fundamental experiments and models, experiments need to be developed that are more efficient and representative of a device's actual operation. Methods are

needed for quickly preparing, manipulating and testing actual nanoscale device components in environments and under loading conditions similar to those it will experience in an application, so as to assess performance and reliability.

The development and validation of more accurate experiments and models, and the dissemination of reliable nanomechanical test methods for material characterization and development, will require the combined efforts of government, industry, academia and national laboratories. Keys to successful collaboration are the standardization and validation of both experimental and modeling methods. Instruments must have traceable calibration paths and standard test methods, so that data from different machines and laboratories can be directly compared. Modeling methods must also be validated, both by comparison to experiments and through the use of standard reference models for which the correct results are well known.

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4. Instrumentation and Metrology for Nanoelectronics, Nanophotonics, and Nanomagnetics

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SCOPE

New nanoscale devices and structures are expected to revolutionize the fields of nanoelectronics, nanophotonics and nanomagnetics. Realizing these advances will require accelerated development of the metrology and instrumentation needed to make reliable, reproducible measurements of device performance and materials' properties and to successfully incorporate devices into commercial products. This area focuses on new metrologies as well as improvements to commercial instrumentation in the areas of nanoelectronics, nanophotonics, and nanomagnetics. Relevant technology applications include advanced semiconductor devices; nanowires, molecular electronics and other "beyond CMOS (complementary metal oxide semiconductor)" technologies; quantum dots, photonic crystals, and other nanophotonic materials and structures; nanoengineered magnetic sensors, magnetic storage, and media; and spin electronics.

VISION FOR NANOELECTRONICS, NANOPHOTONICS AND NANOMAGNETICS

Vision for Nanoelectronics, Nanophotonics and Nanomagnetics

The vision for the future is to successfully develop instrumentation and metrology capabilities for analysis of atomic-scale physical properties, and methods to correlate these properties with nanoelectronic, nanophotonic, and nanomagnetic materials, devices and system performance.

In the future, it is envisioned that instrumentation and metrology will be available to support design, modeling, synthesis and fabrication of advanced materials, processes, and devices for a wide variety of nanoelectronic, nanophotonic and nanomagnetic applications. Capabilities will include nanoscale 3D imaging, chemical analysis, dimensional measurements, *in vivo* analysis during device operation, and material manipulation in hard and soft materials. Multifunctional coupling devices will be available to link nanoelectronics, nanophotonics and nanomagnetics, including nanoscale signal storage and processing.

Metrology developments will enable industry to accelerate discovery and use of new phenomena in materials, structures, and devices with nanometer critical dimensions where interface interactions (rather than bulk atomic behavior) dictate the collective electronic, magnetic, and photonic behavior of the structure or device. Improved resolution of measurement tools by orders of magnitude over current capabilities will make it possible to probe local behavior on the atomic and molecular scale and correlate it with the macroscopic behavior of larger entities.

In *nanoelectronics*, the vision includes tools that (1) can measure statistically significant information for manufacturing (e.g., average of multiple variations) and (2) are capable of point-by-point characterization (e.g., single variations). Of particular importance are physical and

electrical measurements that ensure optimal nanoelectronics system operation, correlation of physical characterization with the electrical properties of the device, fast, non-invasive subsurface/volumetric measurement capability, and 3D-resolved, nondestructive evaluation (NDE) of the chemical, physical, electrical, optical, and other properties with nanometer resolution capability.

In nanophotonics, the vision includes (1) instrumentation and metrology to support the development and seamless integration of nanophotonic materials, components, and devices into photonic, electronic, and hybrid circuits, and (2) advanced nanophotonic-based characterization tools (such as NSOM) for nanoscale 3D imaging and spectroscopic chemical analysis. An important component of both is the accelerated development of improved modeling capabilities that are critical to designing, engineering, and fabricating nanophotonic structures and interpreting probe–sample interactions in nanophotonic characterization techniques.

International Technology Roadmap for Semiconductors

The semiconductor industry has established the International Technology Roadmap for Semiconductors (ITRS), with new sections covering single electron transistors and molecular electronics. Within the ITRS the Metrology Roadmap section discusses the metrology and materials characterization needs for advanced non-classical CMOS and beyond CMOS, including emerging device technology. In 2005 the ITRS includes Emerging Research Materials, along with a section describing the metrology and characterization needs for these materials.

In nanomagnetics, metrology is envisioned to fabricate magnetic structures with 1 nm to 10 nm dimensions, measure their chemistry and structure, measure the magnetization vector of each atom and nanoparticle in these structures and their interactions, and image magnetic domain structure at 1 nm resolution at high speed. Modeling methods will handle multisided scales ranging from 1 nm to 1 m. Measurements will be done in actual operating environments and at picosecond timescales. Magnetization reversal by domain processes or spin rotation methods would be observable, enabling engineering of devices for high-speed switching and sensing. This would affect a diversity of applications from biomedical detection and remediation to magnetic random access memory (MRAM), strategic sensing and homeland security.

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

Reliable, accurate instrumentation and metrology is a critical factor in the successful fabrication and operation of nanoelectronic, nanophotonic, and nanomagnetic devices and systems, such as those in computers and communication systems. Although many advances have been made, much of the currently available metrology techniques are operating at the limits of resolution.

Nanoelectronics

Smaller, faster, denser, and cheaper electronic products have helped propel the information revolution, resulting in faster U.S. economic growth, greater productivity, and the creation of high-tech, high-wage jobs. Nanotechnology applied to electronics is fueling ever-greater breakthroughs and advances in technology for telecommunications, computing, and a host of other applications. Continued advances in nanoelectronics will be driven by innovation and breakthroughs in R&D, whether in industry, government, or academia. The semiconductor industry has been particularly successful in creating synergy and impact in nanoelectronics by organizing and driving the ITRS process. Other emerging nanoelectronic communities could profit from the focus and vision that roadmaps provide.

Current manufacturing practices for silicon technology already reside in the realm of nanoelectronics and future silicon technologies will drive device dimensions below 10 nm. Beyond the 15-year time frame, nontraditional technologies, such as molecular electronics, are expected to play an important role in future electronic systems by complementing the capabilities of nanoscale silicon technology. Current silicon technology, with physical gate lengths below 50 nm, and future devices at the scaling limit will be ultrananoelectronic devices. Materials now have dimensions in the 1 nm to 50 nm range (channel length, film thickness, junction depth, dielectric thickness, and silicon layer thickness in SOI [silicon-on-insulator] wafers). Having a small scale alters material properties (e.g., quantum confinement changes properties), and manufacturing processes increasingly must be controlled at the atomic scale.

Use of MEMS (microelectromechanical systems) and NEMS (nanoelectromechanical systems) technology for metrology sensors and structures is paving the way for next-generation metrology.

Requirements for new field effect transistor (FET) and interconnect materials, device design, and interconnect complexity, and changes in device paradigms based on molecular materials and quantum computing, are stimulating the emergence of new approaches to materials characterization, and critical assessments of more traditional approaches, such as high-resolution transmission electron microscopy (HRTEM) imaging. Advances in electron lens technology are expected to greatly increase the imaging and characterization capabilities of scanning transmission electron microscopy and enable characterization of nanosized areas. New lens technology should allow location of atoms in three dimensions. Another technology known as local electrode atom probe holds the promise of 3D atomic maps. The inclusion of new materials such as high- and low-k dielectrics for gate stacks and interconnects, strained silicon transport layers, and functional molecules creates a greater challenge. Theory has an important role in this revolution—validating measurements, defining the knowledge base for new characterization approaches, underpinning approaches to metrology, and underpinning device and circuit performance models.

Current scientific and technological advancements in nanoelectronics can be described in two major thrusts: (1) traditional CMOS scaling efforts based on classical silicon devices and (2) efforts related to novel emerging devices and structures, based on either (a) nonclassical silicon devices such as FinFETs and multi-gate resistors or (b) beyond-CMOS technology such as single electron transistors, resonant tunneling diodes, nanowire transistors, and nonsilicon nanotechnologies and molecular technologies (molecular electronics, carbon nanotubes).

Traditional CMOS Scaling Efforts Based on Classical Silicon Devices

Activity focuses on the long-term metrology challenges outlined in the International Technology Roadmap for Semiconductors [1] for technologies beyond the 45 nm node. Many of the short-term metrology challenges continue beyond the 45 nm node as well. 3D chemical analysis including 3D dopant profiling is critical. As active device area dimensions approach the spacing between dopant atoms, device behavior differs substantially from that of prior generations, complicating both process simulation and metrology tools. Measurement of dopant element concentration at the required spatial resolution is not currently possible, making the development of such capability a priority for silicon CMOS manufacturing technology.

Microscopy for nondestructive, production-worthy wafer- and mask-level microscopy is currently not available for dimension measurements of 3D structures, overlay, defect detection, and analysis. Critical dimension (CD) measurement must account for sidewall shape and line edge roughness for gate stacks and Damascene trenches. Advances in short wavelength scatterometry must be achieved to address these issues.

Metrologies for Emerging Novel Device Technologies

Traditional CMOS technology is beginning to show fundamental limits associated with the laws of quantum mechanics and the limitations of fabrication techniques. Silicon-based R&D is of primary interest because of the inherent compatibility with CMOS technology. For instance, to permit the continued increase of the density and speed of ULSI circuits, non-bulk MOSFETs (metal-oxide-silicon field effect transistors) are slated to replace conventional bulk MOSFETs. Not only will new materials such as high-k and metal gate electrodes be used, but the new designs will require significant changes to metrology methods and equipment. Likewise, to continue the rapid pace of increased memory density, flash memories based on nanoscale beyond-CMOS silicon quantum dots surrounded by oxide might be used. Other examples include resonant tunneling devices based on nanoscale layers of silicon and confined silicon used in on-chip photonic or light-emitting devices. *Nonclassical CMOS silicon-based devices* have been made on SOI substrates including the FinFET and multigated transistors down to 5 nm gate length, providing a pathway for CMOS scaling for a least the next 15 years. Research is underway in industry, universities, and government laboratories on these advanced (nonclassical) CMOS technologies.

The common thread through many of these examples is the nanoscale confinement of silicon in one, two, or three dimensions (i.e., confined silicon). The electronic properties of devices based on confined silicon are extremely susceptible to small perturbations in structural, material, and chemical properties such as thickness (in the confined directions), interfacial disorder, composition, impurities, and so forth. The extreme sensitivity of the electronic properties of these devices to their nanoscale physical properties poses a significant challenge to metrology.

Nonsilicon nanotechnology and molecular alternatives, including molecular electronics, carbon nanotubes, quantum devices, and computing beyond the horizon of advanced CMOS are referred to as being beyond CMOS. The ITRS specifically identifies the need for R&D on components based on a variety of nanostructures and molecular electronic strategies that might be used beyond CMOS. These range from single or small aggregates of molecules—molecular electronic devices—to rods and tubes of nanometer size. For example, nanorods and carbon nanotubes have been shown to function as transistors, diodes, lasers, and optical detectors—all functionalities required for the complex device architectures of electronic systems 20 years from now.

Molecular electronics is a promising technology for delivering the device density required of beyond-CMOS generations. Molecular field-effect transistors, reversible molecular switches, molecular negative-differential resistors, and molecular rectifying-diodes have all been discovered and characterized, and a prototype molecular-memory device with a density of 10 Gbits/cm^2 has been produced.

Basic electrical quantities, such as resistance/conductance, and protocols for accurate measurement of these quantities are well understood in large-size (tens of nanometers) conductors. In nanometer-sized wires and through molecules, however, the physical basis for electrical charge transport is quite different, and accurate methods for measurement of these quantities are ill defined. Models, test structures, and measurement protocols for electrical properties of molecules are currently inadequate or non-existent. These form the basis for proper simulation of performance of electronic devices and are critical to the assembly of complex nanoelectronic devices.

Nano-assemblies (rods, tubes, dots) are small numbers of atoms or molecules with electrical, mechanical, and thermal properties that provide useful functionality for construction of nanoscale components of electrical devices. Measurement science and tools for property measurements at this scale are currently inadequate and will need to be expanded to achieve viable utilization of these assemblies as components or as artifacts for standards.

Table 4.1
Current State-of-the-Art Spatial Resolutions for Near-Field Scanning Optical Microscopy (NSOM) and Related Techniques

Technique	Transverse Spatial Resolution	Comments
NSOM via nano-apertured tapered fibers	20 nm to 50 nm (best) 100 nm (typical)	Tips are extremely fragile; significant attenuation of optical signal
Apertureless NSOM	10 nm to 50 nm	Strong coupling of morphology with scattering can obscure true imaging
Tip-enhanced nonlinear optical microscopy	10 nm to 50 nm	Strong coupling of morphology with scattering; also strong electronic/optical coupling of tip with sample
Solid immersion lens microscopy	150 nm to 250 nm	High optical throughput and good depth resolution; contact mode frustrates ability to scan
Tip-enhanced solid immersion lens	≈20 nm (predicted)	Solid immersion lens microscopy variant of tip-enhanced nonlinear optical microscopy

Nanophotonics

Photonics—the science and technology of generating, transporting, manipulating, and detecting light—is an integral part of daily life, enabling everything from modern long-distance communications and digital photography to lasers, light-emitting diode lighting and photodynamic medical treatments for cancer. Reducing the size of photonic components is a key driver to increasing the speed, sensitivity, and functionality of devices and systems. Ultimately, nanophotonic circuits may be able to take over where electrical circuits eventually stop working [2]. Future advances in nanophotonics will lead to a diversity of applications ranging from single photon quantum sources and detectors for quantum communications/cryptography to nano-lasers and biomedical diagnostics and photo therapies. Areas in which nanophotonics are expected to

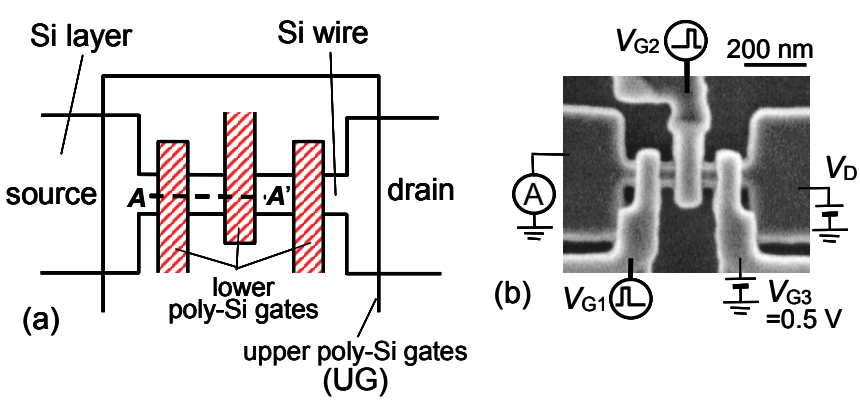

Figure 4.1. Si-wire charge coupled MOSFET device fabricated on a silicon-on-insulator wafer. (a) Schematic top view of the device. (b) Scanning electron microscope image of the device before upper gate formation (courtesy of Akira Fujiwara, NTT Basic Research Laboratories, NTT Corporation; reprinted with permission from [3], © 2004, American Institute of Physics).

have the greatest effect include optical communications and data transmission, quantum cryptography and computing, near-field optical spectroscopy, bioimaging, optical storage and various sensing applications. Extending photonics to nanoscale dimensions requires understanding and exploiting the interaction between light and matter on a scale much smaller than the wavelength of optical radiation. The nanophotonics spectral range of interest is centered on the visible spectrum but extends from vacuum ultraviolet to near infrared, depending on the application. A conceptual framework to describe the current state of nanophotonic advancement involves nanoscale confinement of optical radiation, nanoscale confinement of matter, and nanoscale photo processes [4, 5]. It should be noted that nanophotonics is defined in various ways, and the descriptions below are not intended to provide complete coverage of this field [5].

Nanoscale Confinement of Optical Radiation

The spatial resolution of optical microscopy and spectroscopic analysis of materials and devices using NSOM and related techniques is dramatically improved by probing the optical near-field with subwavelength resolution. Sample features much smaller than the far-field diffraction limit are imaged by probing the evanescent light fields through a subwavelength-sized aperture (20 nm to 200 nm) very close to the sample surface. The size of optical fiber aperture and the distance from tip to the sample (≈5 nm to 50 nm) then control the spatial resolution that can be achieved.

State-of-the-art nanoscale spatial resolutions now achievable with NSOM and related nano-optical techniques have improved greatly over the past 20 years (see Table 4.1). The tip-enhanced NSOM techniques described also take advantage of the benefits of nanoscale confinement of matter (discussed in the next section), in which sharp metallic tips or metallic nanoparticles are used for optical field enhancement and higher spatial resolution. A noteworthy experiment demonstrating the resolution of NSOM is the collection of near-field Raman images of single isolated single-walled carbon nanotubes (SWNTs) with a spatial resolution of ≈25 nm [6].

In addition to images based on changes in light intensity, NSOM can take advantage of many different optical contrast mechanisms including various spectroscopies (reflectivity, transparency, polarization, fluorescence, photoluminescence, Raman, and others). To illustrate the broad applicability of NSOM, Figure 4.2 demonstrates high-resolution polarimetric NSOM images of polymer (isotactic polystyrene) crystallites in a 15 nm thick film. A more complete discussion of nanocharacterization methods including other scanned probe microscopies is found in Chapter 2 of this report. Additional non-optical nanocharacterization methods relevant to nanophotonic characterization not discussed here include scanning Kelvin, capacitance, and photovoltage microscopies.

NSOM via nano-apertured metal-coated tapered optical fibers is used in either excitation mode or collection mode. Spectroscopic techniques include photoluminescence (excitation or collection), Raman (collection), and multiphoton spectroscopy (collection). This method has several shortcomings. For example, the aperture at the end of the metal-coated tapered fiber tip cannot tolerate much optical power before failure. For this reason, NSOM is a poor method for excitation-mode spectroscopy. In collection mode, the tapered fiber tips attenuate most of the light, resulting in long data acquisition times. The metal-coated apertured tips are very fragile, and because NSOM collection mode is usually done simultaneously with AFM, aperture failure may occur during mechanical contact with the surface during a scan. As a result, the tip's transmission characteristics may change during a scan, increasing optical throughput at the expense of transverse spatial resolution.

Apertureless NSOM allows scanning of a probe tip. The approach is to scatter light off a small metal probe (typically an STM tip) and render a high-resolution optical image of the surface. No

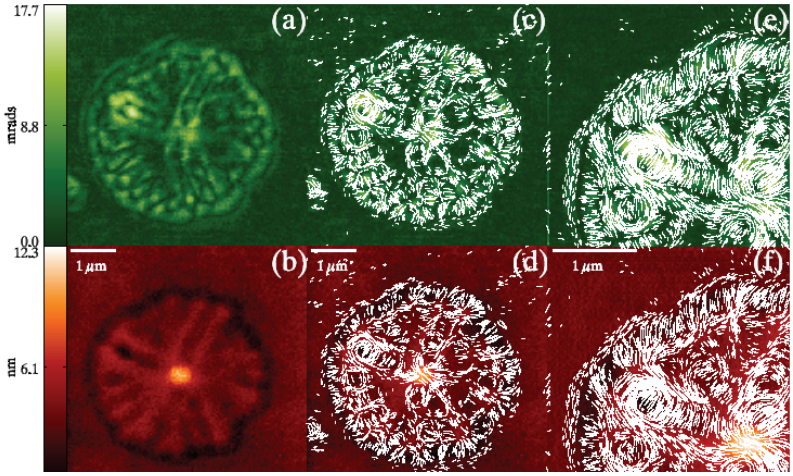

Figure 4.2. Retardance, fast axis alignment, and topography images of polymer (isotactic polystyrene) crystallites in a 15 nm thick film. Images taken simultaneously with a polarimetric NSOM (a) retardance; (b) topography; (c) retardance with overlaid fast axis orientation; (d) topography with overlaid fast axis orientation; (e, f) higher-resolution scan of upper-left quadrant (courtesy of Lori Goldner, NIST; reprinted with permission from [7]).

tapered fiber or nanoscale aperture is involved. Excitation occurs via a number of methods including simple illumination, photo-luminescence, and so on. Collection and detection is performed in the far field. The interpretation of the data is difficult because of the complications imposed by the coupling of the topography and the optical scattering from the tip.

Tip-enhanced apertureless NSOM, also called tip-enhanced nonlinear optical microscopy, is related to apertureless NSOM. With this method the added effects of optical field enhancement of plasmon modes excited in small metal tips (usually Ag or Au) improves the contrast of light emitted from the surface over that obtained via scattering alone. In certain instances, this enables a transverse spatial resolution in the range of ≈10 nm with mapping luminescence generated by two-photon excitation. Drawbacks include strong tip-sample interaction that may quench luminescence.

Solid immersion lens microscopy (SIL) and variants employ high-numerical aperture optics to enhance the resolution within the diffraction limit rather than operating below the diffraction limit, as is done with scanning tip/probe methods. To get the best resolution with SIL, a high-numerical aperture optic is placed in hard contact with the surface of the sample. SIL has the advantage of some depth resolution (not possible with other methods) for subsurface imaging. This has been demonstrated using the numerical aperture increasing lens (NAIL) technique to achieve 230 nm resolution (at 1050 nm wavelength) for near-infrared inspection of buried Si integrated circuits [8, 9]. A significant disadvantage of SIL is the need for hard contact, which complicates scanning. Some SIL variants actually employ an air gap between the high-numerical aperture optic and the sample that enables scanning but at the expense of resolution.

Tip-enhanced solid immersion lens is another SIL improvement under discussion that uses optical field enhancement at the SIL focus via metal nanoparticles, in a similar manner as that used in tip-enhanced apertureless NSOM. This improved SIL is expected to yield transverse spatial resolution of roughly 20 nm. All of the scanning probe and tip-enhanced optical techniques described above experience inescapable, strong tip-sample interactions. To accurately define a "resolution limit" for the quantity to be measured, models must be available to deconvolve tip-sample interactions for the measurement employed. The usefulness of modeling in this capacity has already been demonstrated [10].

Nanoscale Confinement of Matter (Quantum Dots, Photonic Crystals, Plasmonics, Others)

A very active research area in nanophotonics is the development of nanomaterials and nanostructures used to control the generation, propagation and detection of light. The successful

development of low-dimensional inorganic semiconductor structures (quantum wells, quantum dots, quantum wires) with unique optoelectronic properties that are controlled by strain, size, size distribution, and other properties is well known. Recent progress has been made in the development of "single-photon-on-demand" sources based on quantum dots and complementary single-photon-resolving detectors that together will significantly affect quantum communications/cryptography applications. New biological applications of these structures are being actively pursued.

Significant advances over many years have been made in the development of photonic bandgap crystals [11, 12], a major thrust area in nanophotonics research worldwide. Significant theoretical research effort has gone into designing and modeling photonic crystal structures using frequency and time domain techniques to calculate accurate band structures, which allow prediction of optical properties and performance engineering of photonic crystal optical circuitry.

Plasmonics is a growing field in which metallic nanostructures and nanoparticles with unique electronic and photonic properties are being developed for applications such as guiding light over extended distances with lateral dimensions much less than the wavelength. Research is also active in nanophotonic nanocomposites—media with randomly distributed nanometer size domains in which different domains can be separately designed and optimized to serve different photonic functions [5].

Nanoscale Photoprocesses

Significant progress has been made in the development of nanoscale photoprocesses, particularly for nanofabrication of complex 3D structures for photonic devices, laser nanofabrication, optical memory, micromachines, and integrated optical waveguides with high spatial resolution [13]. Although current optical storage technologies achieve 1 Gb/cm^2, there is potential to reach 80 Gb/cm^2 in the future. Volume holographic storage, optical spectral hole-burning holography, and two-photon optical memories are also being explored.

Nanomagnetics

Magnetism is found all around us, hidden inside many of the devices and products we use every day. Magnets are key components in motors, transformers, and generators and are the basis for magnetic recording media and magnetic writing and sensing devices. They are found in credit cards, all electronic devices, cellular telephones, microwave devices, xerographic copiers, theft-control devices, airport security systems, and magnetic resonance imaging (MRI) contrast agents. The characteristics of the magnet (e.g., susceptibility, hysteresis, coercivity, anisotropy, saturation magnetization, squareness, switching speeds) are different for each application. As the size of the magnet decreases, measuring these characteristics becomes more difficult, as the magnetic field emanating from or the force exerted by that field decreases.

Experimental Nanomagnetic Measurement Techniques

There are currently many experimental techniques available for making magnetic measurements. These include magnetometers (e.g., superconducting quantum interface device (SQUID), vibrating sample magnetometer (VSM), and alternating gradient magnetometer (AGM)) for making magnetic moment measurements in relatively static conditions, susceptometers and B-H loopers for measuring magnetic susceptibility and hysteresis loops at different frequencies, domain imaging methods (e.g., SEMPA, MOIF, MFM, Kerr, Lorentz Microscopy), techniques for probing spin dynamics (e.g., FMR, pulsed fields on striplines), and magnetic spin orientation detection tools (e.g., neutron diffraction, neutron reflectivity, Mössbauer effect). Techniques are also available for modeling magnetic spins in materials (e.g., micromagnetic, finite element). The magnetic

characterization tools currently available (VSM and AGM) measure the magnetic properties of a large collection of particles. Our ability to extract information from these measurements is limited by the need to account for the unknown distribution of magnetic properties.

Today's magnetic characterization tools either have very high sensitivity or high-speed capabilities, but not usually both. Similarly, high-resolution imaging tools are also slow or have low resolution but high speed. Some of these tools are capable of making absolute measurements (as there are standard reference materials or procedures available), but most are capable of only relative measurements.

Magnetic Recording

The areal density in magnetic recording has surpassed ≈11 Gbit/cm^2 (≈70 Gbit/in^2) in products and ≈17 Gbit/cm^2 (≈110 Gbit/in^2) in laboratory demonstrations. At 15 Gbit/cm^2 (100 Gbit/in^2), the bit dimensions are about 35 nm (bit length) and 200 nm (track pitch, bit width) translating into data rates beyond gigabits per second for high-end drives. A higher magnetic moment write head will create a higher field, H, which is becoming more necessary as the coercivity, H_C, of smaller media gets higher. Higher coercivity and control of anisotropy (crystal structure, grain morphology, shape, defect created, material composition) are needed in higher-density media to overcome the superparamagnetic limit. The metrology required for controlling these parameters at the nanometer-size scale of these features is largely nonexistent. With the reduction in bit dimensions, maximum magnetic domain sizes in heads and media have also been reduced in size to below the resolution of most observation techniques, resulting in their having to be calculated from computer models that need verification. Consequently, magnetic switching mechanisms at these small-size scales are only assumed.

Spin Electronics (Including Magnetic Random Access Memory, MRAM)

Magnetic random access memory (MRAM) is advantageous over conventional RAM because it offers "instant on" and "instant off" capability. Information is still maintained in the storage device, even though there is no power provided to it. This is different from other types of RAM, which require an electric potential to be maintained. It also promises read/write times faster than 10 ns, unlimited endurance (>10^{12} reversals), switching energies less than 0.1 nJ, and high scalability. The material concerns are the same as those for regular hard disk media, and hard disk materials capabilities are much further advanced. The control of magnetization dynamics and the material's nonuniformity are current limitations to MRAM. Also, much better sensitivities and signal-to-noise ratios would be possible if spin valve structures with giant magnetoresistance values greater than 30% could be achieved. In 10 years, the thermal properties of the MRAM will also become important as MRAM devices will need to take advantage of heat-assisted recording.

Permanent Magnets, Soft Ferromagnets, Biomedical, Sensing

Composites of nanometer-thin materials have vastly different magnetic characteristics from bulk magnetic materials and can be used to create permanent magnets with much higher energy densities (e.g., composites of hard ferromagnets with soft ferromagnets) as well as very soft ferromagnets (e.g., soft ferromagnets with nanometer-sized grains). Nanometer-sized ferromagnets dispersed in a paramagnetic or diamagnetic material have also been shown to possess enhanced magnetocaloric effects, which could lead to room-temperature magnetic refrigerators, a highly efficient potential cooling technology. For all these applications methods need to be developed for inexpensively and controllably preparing such materials in large enough sizes and quantities.

Magnetic nanoparticles have potential biomedical applications including detection of biomolecules, separation of biomolecules and cells, MRI contrast enhancement, and hyperthermia therapy.

Beyond detection of biomolecules is the use of magnetic fields to manipulate biological processes. One approach is to selectively bind a magnetic particle to a site and to use an AC magnetic field to heat the particle and the region around the particle, thereby influencing a biological process (e.g., denaturing a protein or killing a cell). To develop this science it would be useful to manipulate individual nanoparticles so as to place them precisely in a desired location, perhaps by a magnetic field. To do this, large magnetic field gradients will be required; present capabilities do not yet allow control of individual nanoparticles. Consequently, micrometer-sized particles are now being used.

GOALS, BARRIERS, AND SOLUTIONS

Nanoelectronics

The vision for nanoelectronic metrology is aligned with industry goals and roadmaps that support the continual advances of CMOS technologies as well as R&D beyond CMOS technologies. The overall goal for the future is to have available instrumentation and methods for analysis of atomic-scale physical, electrical, and chemical properties. The path from laboratory prototype to robust manufacturability depends significantly on yet-to-be-developed metrology tools having physical, chemical, and electrical characterization capabilities. Methods to correlate these properties with nanoelectronic device and system performance will be critically needed. The barriers are formidable and relate to developing and characterizing new unproven materials as well as developing ways to theoretically model and simulate the properties and the corresponding device behavior. Some of the specific barriers and priority needs for nanoelectronics are outlined in Table 4.2.

The area of nanoelectronics has benefited tremendously from the ITRS [1], which is regularly reviewed and updated by experts from around the world. The ITRS describes challenges in a dozen fields as well as supporting metrology and ties progress to key technology nodes that follow Moore's Law and other trends. The benefits of achieving the ITRS technology targets are readily apparent. By the end of the decade, the cost of equivalent memory will significantly decline, and microprocessors will be much faster.

The ITRS projects that progress will stall without research breakthroughs in most technical areas. Fundamental limits of the materials used in the current planar CMOS process, the process that has been the basis for the semiconductor industry for the past 30 years, are being reached. Further improvements in the current planar CMOS process can continue for the next 5–10 years by introducing new materials into the basic CMOS structure. It becomes evident that even with the introduction of new materials, most of the known technological capabilities of conventional planar CMOS device structures will approach or have reached their limits.

However, as the ITRS looks forward, the use of SOI wafers and advanced device designs will extend MOSFETs (a.k.a., advanced CMOS) for at least 10–15 years. To provide a more cost-effective alternative to current planar CMOS, a considerable amount of research into this advanced CMOS, beyond-CMOS technology is necessary. The trend toward new materials will continue. The decrease in dimensions of interconnection between devices will also require new materials and structures. This will create new challenges for metrology.

Table 4.2
Challenges and Barriers in Nanoelectronic Instrumentation and Metrology

Instrumentation	Accuracy
Chemical Composition	• Two-dimensional/3D dopant profile characterization for CMOS/beyond CMOS nanodevices • 3D atomic mapping for materials characterization • High purity organic raw materials (Now: 10^{-3}; In less than 5 years: 10^{-5}) • Chemical bonding information
Electrical Properties	• Identifying critical long-term reliability failure points within nanodevices (i.e., new failure mechanisms) • Low-power, non-destructive methods for I-V, C-V electrical characterization; aF, fA • RF at low cost for high-frequency applications (GHz) • Electronic structures of buried organic/inorganic interfaces • Electronic properties of materials/interfaces within device structure at nanometer scale • Mobility measurements for materials, devices, and structures
Thermal Properties	• Thermal mapping for nanoelectronic structures • Improved temperature conductivity migration/diffusion characterization
Structure and Mechanical Properties	• Robust devices to measure nanoscale mechanical properties in 3D integrated structures • Standards for materials, measurements, and devices to evaluate and benchmark performance • Statistically significant detection of defects in high aspect ratio structures • Statistically significant measurements for physical and electrical properties during manufacturing • Improved nanometer-level positioning abilities for optimum nanoassembly • "Critical Dimension" metrology for manufacturing <10 nm; nondestructive, 3D at high speed • Stress measurement in nano-sized areas
Optical Properties	• 3D measurement capability of complex index of refraction • 3D optical spectral mapping capability • Ability to better measure resist performance in small nanostructures
Modeling and Simulation	• Measurement of model input parameters • Current state of the art: independent characterization of contacts, "bulk" interface; future need: as device volume decreases, need to extract these quantities from same measurement • All metrology is based on manufacturing models and therefore needs improved "rigorous" solvers and "reduced order" models • Current state of the art: apply multiple techniques; expert analysis to merge; future need: merged techniques ("multispectral") and more automated analysis • Greater knowledge of relationship between nanoscale physical and chemical properties and final electrical device performance

Table 4.3
Key Challenges and Barriers for Nanophotonics

Metrology	Challenges/Barriers
Materials and Devices	• Hierarchical multiscale modeling methods to correlate measured physical and device electrical properties • Tools with relevant sensitivity, resolution, and precision • Standardized methods/models for analysis of metrology data • Improved contacts and electrical characterization • Reliable, robust test platforms
Techniques and Instrumentation	• Integration of devices and material with different functionalities • Difficulty in acquiring sufficient information to obtain desired properties in sub-surface imaging • Components and devices to improve signal

The ITRS outlines technical barriers that must be overcome in both the near and long term, including many that affect metrology. For example, requirements for optical lithography after 2010 will necessitate the introduction of next-generation lithography tools such as immersion optical lithography, extreme ultraviolet lithography, and electron projection lithography. Breakthroughs are also needed to interconnect all of the transistors required on a single chip and might include optical or wireless connections rather than conventional electronic/metal interconnects. Finding solutions to these challenges will require increased understanding of fundamental device physics and material properties, as well as new approaches to technical problems and concentrated efforts to develop metrology breakthroughs.

New physical limitations of performance (tunneling, quantum state confinement effects) and size emerging from the aggressive scaling of traditional CMOS devices will create new metrology challenges. Requirements for new FET and interconnect materials, device design, and interconnect complexity will bring further challenges. Additional changes in device paradigms that are based on molecular materials and quantum computing will create additional concerns. "Beyond classical" CMOS manufacturing techniques (<10 nm gate) will be a critical need. Metrology applied to IC (integrated circuits) will require a rapid infusion of new ideas with more aggressive scaling of CMOS devices incorporating new materials, and new device structures based on materials both inside and outside the vision of the ITRS.

Interconnect issues remain a significant challenge. The molecule–metal contact is an integral component of the device's performance. This junction can make a molecular wire conduct well or poorly; it can make a molecule rectify or not. The functional electrical properties of the molecule or nano-component are perturbed by the presence of the conducting connection. New measurement capabilities will be required to permit effective deployment of practical devices based on nano-components.

Future needs in nanoelectronics include the ability to generate a nanoscale, buried two-dimensional (2D) species map. Current capabilities are at the 100 nm scale; a 5 nm scale is sought in less than 5 years. Tools will be needed to measure and map electrical defects (e.g., state in gap, local charge) in small (one dimensional) structures, and to measure the location of atoms in 1–50 nm^3 volumes.

Structural metrology needs include interface characterization for inorganic/organic nanostructures and tools to conduct 3D-resolved, NDE analysis of all properties in buried structures with nanometer resolution. Test structures are needed to measure electronic properties related to

individual nanoelectronic devices. Standard electrical test methods for reliability of new materials, such as ultrathin gate and capacitor dielectric materials, must be developed. Tools will also be needed for *in situ* characterization of hard/soft/hard interfaces and local structure (hard: inorganic, soft: organic/biological). Nondestructive, production-worthy wafer- and mask-level microscopy is needed for critical dimension measurements of 3D structures, overlay, defect detection, and analysis. CD measurement must account for sidewall shape and line edge roughness for gate stacks and Damascene trenches. Advances in short-wavelength scatterometry must be achieved to address these issues.

Property measurement capabilities are needed to detect localized electronic properties and their relation to device performance. This includes surface/interface chemistry and structure in relation to electronic properties, as well as the ability to relate atom-by-atom structure and chemical bonding information to electrical response. Tools will be needed for accurate molecular property measurements (e.g., electrical, optical, chemical). Greater knowledge of the relationship between nanoscale physical and chemical properties and final electrical device performance is desired. More sensitive nanoscale 2D RAMAN/IR capability is needed, with the goal of moving from current capability of 100 to 5 nm in less than 5 years.

Computational needs include models relating nanoscale physical properties with device performance, and hierarchical computational tools that can focus on different aspects of complex problems (position, time, Ψ). Models can now perform independent characterization of contacts and "bulk" interface. As device volume decreases, there will be a need to extract these quantities from the same measurement. Metrology is based on manufacturing models and will need improved "rigorous" solvers and "reduced order" models.

Statistical limits for sub-65 nm process control need to be developed. Controlling processes where the natural stochastic variation limits metrology will be difficult. Examples are low-dose implant, thin gate dielectrics, and edge roughness of very small structures.

Nanophotonics

There are a number of challenges and barriers to be addressed in nanophotonics (see Table 4.3). Nanophotonics as a field is less mature than nanoelectronics and nanomagnetics and is still seeking a niche where nanophotonics provides unique functionality. Nanophotonics is not as clearly aligned with industry goals and roadmaps as nanoelectronics and nanomagnetics. Although nanophotonics is an active and vigorous field, it would benefit from more focused and integrated challenges and roadmapping activities to encourage broader involvement in common problems faced by researchers, technology developers and users.

In the realm of instrumentation and metrology to support nanophotonics, significant science and technology challenges exist in spectroscopy of nanosystems, photonic crystals, optical nanostructures, optical nanocomponents and hybrid material systems, nonlinear and electro-optic materials, and modeling. Hard problems and potential solutions to address these challenges were analyzed in three critical areas: (1) advancing optical spectroscopy at the nanoscale, including developing enabling components for nanoscale spectroscopy; (2) developing metrology for design, fabrication and integration of optical nanostructures; and (3) modeling nanoscale properties and photonic structures.

With high spatial resolution and the versatility of well-established spectroscopies, nanophotonics-based *optical imaging and spectroscopy* provide significant benefits for nanoscale characterization of material, devices and biological systems. Nanoscale subsurface imaging of buried interfaces has become increasingly important with the growing need to characterize complex multilevel

nanofabricated structures *in situ* and in operation, with the hard problem of imaging through subsurface heterogeneous media between the structures of interest and detectors outside the sample.

Significant additional research is needed to explore new subsurface imaging techniques and extend existing techniques (such as NAIL, described previously) to meet the growing challenges. Nanophotonic-based bioimaging is also needed, with goals of real-time imaging and spectroscopy of the process of transcription *in situ* and multiprobe ~1 μeV nanometer resolution spectroscopy for surface and buried interfaces. Extending the capabilities of optical spectroscopy at the nanoscale will allow measurement of average ensemble performance for biological and other systems through single-element and single-molecule optical measurements.

More generally, there is a significant barrier of inadequate signal, resolution and speed of nanocharacterization techniques (also described in Chapter 2). Extension of nanocharacterization tool performance factors by another factor of 10 or more is needed for the near future, along with accurate, fast positional control; understanding of probe-system perturbations and interactions; reliable and reproducible tip behavior; and compact instrument design. Tools should allow time resolution on femtosecond timescales and full characterization of quantum and nonlinear effects. For nanophotonics-based characterization, enabling components are needed for nanoscale spectroscopy. These include improved sources that are bright, narrow-bandwidth, fast, and low power; improved nanoscale detectors that are hyperspectral, fast, and low cost; and nanooptical probes that are long-lived, bright, pure, and narrow, and with broad bandwidth.

In the area of optical nanostructures and more broadly the *fabrication and integration* of nanophotonic components in electronic, photonic and hybrid circuits, technical barriers include the understanding and control of nano- to meso-morphology in low-dimensional photonic structures. The hard problem is that controllable synthesis methods require improved *in situ* monitoring and control of growth, as well as *in situ* techniques for optical characterization and the ability to link morphology to performance. Proposed solutions include development of extensive optical databases for III–V semiconductors as a function of temperature (particularly at elevated temperatures), composition, stress, and processing conditions.

A critical pervasive need in nanophotonics is accurate *modeling of nanoscale optical properties* and the interaction of light with nanostructures. Although computationally demanding, nanophotonic modeling is essential to design and optimization of nanophotonic structures for commercial applications and for interpretation of nanophotonic-based microscopy/spectroscopy results. One hard problem is increasing the availability of reliable materials data as input to models. In addition, realistic modeling of probe–sample interactions in nanophotonic spectroscopy techniques such as NSOM presents a challenge. Solutions include accelerated development of user-friendly PC-based modeling tools and visualization environments. There is also a need for computational techniques with improved efficiency and usability, including more recently developed finite-difference time-domain method and beam propagation methods.

Nanomagnetics

Challenges for nanomagnetics can be categorized in terms of magnetic recording and spin electronics. Figure 4.3 illustrates some of the challenges in magnetic recording. Table 4.4 summarizes the key challenges and barriers for nanomagnetics in both these areas.

Magnetic Recording

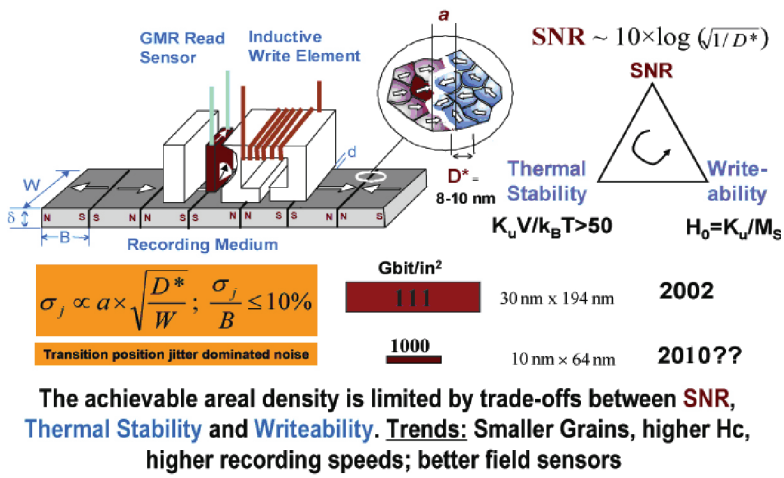

Figure 4.3. Challenges in magnetic recording (courtesy of Dieter Weller, Seagate Group).

A number of unique magnetic metrology needs arise because of the perpendicular magnetization orientation and presence of a soft magnetic underlayer in the media. In addition to the general need for larger fields in magnetometry (media anisotropy fields > 2 T) there is a need to decouple hard from soft materials, favoring magneto-optical methods because of the relatively shallow penetration depths of optical beams in metals. It is also necessary to correct for demagnetization effects and to characterize magnetic dispersions resulting from segregation variations and variations in the exchange coupling from grain to grain. Practical magnetic imaging on the sub-10 nm length scale is critical to improving the microscopic understanding of bit transitions, which depend not only on media nanostructural parameters (grain size, dispersions, exchange, cluster size) but also on the recording process (write field gradient, affecting media transition parameter).

An important challenge is to directly measure and map the head field at the timescale of 100 ps to 1 ns. At such speeds, precessional effects dominate and real-time, pump probe techniques are being explored. It is argued that angled fields can reduce the switching coercivity by up to a factor of two (45°, Stoner Wohlfarth switching astroid), and several novel write head designs (trailing pole, Mallary head) and media designs (tilted anisotropy media, Gao and Bertram) have been proposed to accomplish that. Most are based on micromagnetic model calculations, which need to be founded in experiments. Going much beyond 0.15 Tbit/cm^2 (~1 Tbit/in^2) will require more drastic changes of heads and media (Fig. 4.3). One of the fundamental limitations relates to the media sputter fabrication process, which may not allow the tight grain size and magnetic dispersions required in models. So-called self-organized magnetic arrays (SOMA) of chemically synthesized FePt nanoparticles are therefore being explored as alternatives. These structures show extremely tight size distributions (<5%) and are magnetically much harder than current co-alloys.

Key challenges are control of the crystal structure, magnetic easy axis, avoidance of sintering in FePt (annealing requirement of about 700°C can lead to grain growth), and establishing large-scale ordering and registry on the length scale of a disc. In addition, writing will require temporal heating and cooling in a magnetic field (HAMR—heat-assisted magnetic recording), which sets the stage for a host of needs in heads (heat and field delivery schemes at sub-25 nm dimensions), media (e.g., thermal property control, temperature-dependent magnetics, Curie temperatures, blocking temperatures), and head disc interface (e.g., heat-resistant new lubricants and wear/corrosion resistant materials).

It is envisioned that eventually a combination of SOMA and HAMR may lead to single particle per bit recording, with ultimate densities near 8 Tbit/cm^2 (50 Tbit/in^2) (10 years storage time, ambient temperature, FePt type anisotropies). Some form of assisted self-assembly, (e.g., via topographic or chemical prepatterning on the micrometer length scale) would be required to coat discs with the required uniformity. A single particle (bit) in this scenario has lateral dimensions of about 3 nm,

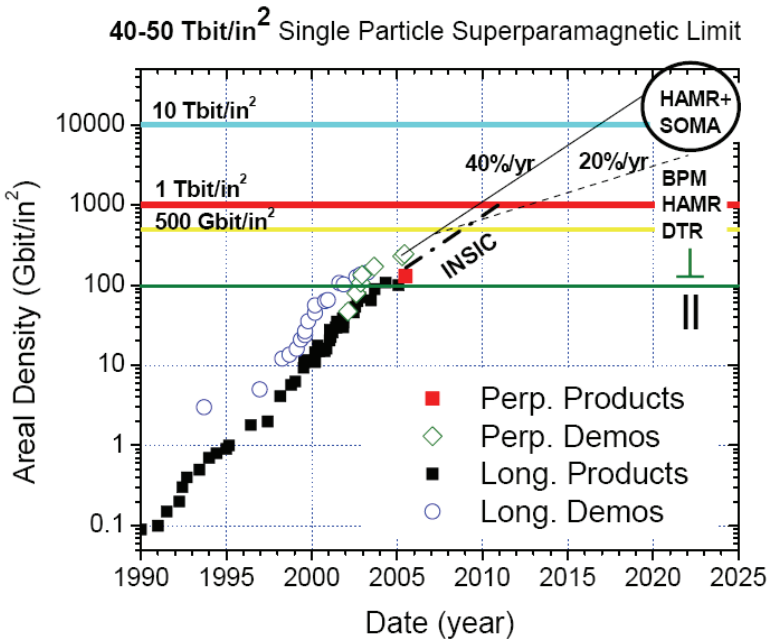

Figure 4.4. Potential roadmap for single particle per bit recording with densities near 8 Tbit/cm^2 (courtesy of Dieter Weller, Seagate Group; data updated post-workshop, 2006).

corresponding to the single particle superparamagnetic limit for FePt, and comprises only about 1,000 atoms, with a large fraction of atoms occupying the surface.

It will be critical to develop magnetic measurements and structural tools to characterize such small magnetic units and their surfaces, which may be quite different from bulk and be strongly dependent on the chemical environment.

A tentative roadmap based on this strategy is shown in Figure 4.4. Significant progress in materials and property control on the nanometer length scale will be necessary to enable what is believed to be ultimately possible in magnetic recording. The time scale for reaching the single particle super-paramagnetic limit is estimated to be at least 10 to 20 years. Metrology will play a key role in reaching this goal.

High-resolution energy dispersive X-ray (EDX) analysis on films containing as-prepared FePt nanoparticles revealed a distribution of particle compositions. Although the chemistry to prepare FePt nanoparticles gives narrow particle size distributions, composition varies from particle to particle. Measuring the composition of individual particles having diameters of 3–4 nm is very difficult, generally beyond current capability. Routine methods are needed to determine the composition of individual nanoparticles so that the chemists can prepare particles with narrow composition distributions (<15% dispersion).

The ultimate of high-density limit magnetic recording technology is expected to be recording in monolayer films of $L1_0$ phase FePt nanoparticles, where one bit is recorded in a single particle. This presents many challenging problems. The most fundamental problems are how to record and read at this extreme spatial resolution. It is known that a distribution of particle compositions is present, and the sintering that occurs during heat treatment gives rise to a distribution of particle volumes.

The magnetic characterization tools currently available to us (VSM and AGM) measure the magnetic properties of a large collection of particles. Our ability to extract information from these measurements is limited by the need to account for the unknown distribution of magnetic properties. Accordingly the metrology challenge is how to measure and control the structure and magnetic properties of individual particles. In addition, elimination of particle agglomeration and control of grain growth will remain a serious metrology issue.

Spin Electronics

For hard disk read heads and MRAM, the challenges are in understanding the behavior of magnetoresistive devices on the 10 nm length scale. Specifically, there are issues of curling, vortex, layer–layer coupling, effects of defects and roughness, interfaces, and temperature. In all cases, the

relevant timescale for magnetic spin reversal will shrink below 1 ns. For MRAM in particular, reducing the energy required to switch and read bits will mean better control of switching sequences on the 100 ps time frame. The need for new spintronics devices in these industries will occur when the means of storing and reading information are not viable at the relevant dimensions. The most immediate hurdle appears to be the thermal stability of magnetic objects on the 10 nm scale, and several development efforts are addressing this. If these are successful, it is likely that the present modes of devices can be extended to the 10 nm scale.

Table 4.4
Key Challenges Barriers for Nanomagnetics

Metrology	Challenges/Barriers
Measurement of Magnetic Properties	• Ability to manipulate samples at the nanoscale • Lack of magnetic sensitivity • Lack of spatial resolution • Theory for measurement analysis to bridge magnetic theory with phenomena • Measurement accessibility (cost, size, portability of appropriate measurement techniques) • Lack of appropriate samples for measurement; *in situ* measurement capabilities
Imaging of Spin Dynamics	• Ability to manipulate samples on nanoscale • Accurate modeling of detection probe—analytical and micromagnetic theory • Fast and large bandwidth electronics for fast data acquisition • Delivery of fast, spatially localized, high-field pulses • High sensitivity detectors; scanning probe response time • X-ray optics
Measurement of Spin Transport	• Lack of ferromagnetic semiconductors with Curie temperatures well above 300 K • Probes that decouple the measurement from the phenomenon • Method for measuring a single electron spin—only way of getting it now is via creation and measurement of a photon • Lack of a generator of a single electron spin • Probe for measuring magnetoresistance locally

The important measurement to make in spin electronics devices is spin current. Spin accumulation is generally measured and spin current is then derived. In addition, spin accumulation can only be measured in some materials (e.g., in GaAs). In some important materials (e.g., Si) even spin accumulation has not been measured.

With this technology path in mind, a proposed nanometrology challenge for nanomagnetics is to (a) measure the magnetic field with 0.25 nm spatial resolution (on-wafer if possible), (b) measure the magnetic moment of 100 × 100 × 100 atoms, (c) predict the internal moment orientation structure, perhaps using scanning instruments, and (d) measure these properties on a 10 ps timescale.

R&D INVESTMENT AND PRIORITY RESEARCH NEEDS

Nanoelectronics

Priority R&D topics for nanoelectronics are organized into two areas below and described in detail in Priority Topics 4.1 and 4.2.

- *Nanoelectronics instrumentation and metrology for advanced non-classical CMOS:* methods and instrumentation for analysis of atomic-scale physical, electrical, and chemical properties, and methods to correlate these properties with nanoelectronic device and system performance.
- *Nanoelectronics instrumentation and metrology for emerging novel devices beyond CMOS:* instrumentation and methods for analysis of atomic-scale physical, electrical, and chemical properties, as well as methods to correlate these properties with novel, emerging device (beyond CMOS) and system performance.

Nanophotonics

In nanophotonics, priority research needs were identified for the two categories summarized below. These are described in more detail in Priority Topics 4.3 and 4.4.

- *Materials and devices for nanophotonics:* design, modeling, and synthesis of nanophotonic materials and devices for optoelectronics, data storage, light sources, sensors, optical computing, and biomedical instrumentation. This includes optimizing growth of low-dimensional structures and heterogeneous systems and measuring materials properties under practical deposition and operating conditions.
- *Techniques and instrumentation for nanophotonics:* concurrent improvements in speed, signal and resolution of nanophotonic measurement techniques and instrumentation are needed for nanoscale 3D imaging, chemical analysis, and material manipulation in hard and soft materials. In particular, advanced nanophotonic techniques for subsurface imaging and 3D tomography are a priority to allow noninvasive and nondestructive device imaging.

Nanomagnetics

In nanomagnetics, priority R&D needs were identified in three major areas of instrumentation and metrology, summarized below and described in more detail in Priority Topics 4.5–4.7.

- *Measurement of magnetic properties of a cubic nanometer of material:* develop the means to measure the magnetic moment of an individual nanoparticle (which would mean about 500 Fe atoms and a magnetization of about 10-20 $A \cdot m^2$ [10-17 EMU]), to measure the moment and its interactions on an atomic length scale, and the ability to scale up the measurement to large ensembles. It would enable the measurement of buried layers, would require the application of a magnetic field to a very small volume (≈ 1 nm^3), and would enable imaging the magnetic polarization in 1 nm^3 of material.
- *Imaging of spin dynamics:* develop tools for 3D imaging, for subnanometer resolution, and for subnanosecond data collection (not averaged over many cycles) and would have element specificity.
- *Measurement of spin transport in materials:* develop models to infer spin polarization current from spin accumulation measurements, tools for measuring spin directly, and probes for decoupling the measurement from the phenomena. None of these capabilities presently exist. It will also be necessary to develop tools to operate in the industrial environment.

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

The nature of nanotechnology dictates the need for interdisciplinary groups to investigate and develop the field. The NSF NIRT (Nanoscale Interdisciplinary Research Teams) program is a good example. Instrumentation and metrology also tend to limit the development of new areas, and in the area of nanotechnology, this is particularly expected to be true. To probe the nanotechnology

Priority Topic 4.1. Nanoelectronics Challenge
Instrumentation & Metrology for Advanced CMOS

VISION AND GOALS The goal for the future is to have available instrumentation and methods for analysis of atomic scale physical, electrical, and chemical, properties, as well as methods to correlate these properties with nanoelectronic device and system performance for advanced CMOS.

KEY PERFORMANCE REQUIREMENTS

- Spatially resolved characterization (1, 2 and 3D as appropriate) of physical properties, ranging from nanometer to atomic scale
- Physical properties of interest include chemical composition, electrical, thermal, structural, mechanical and optical properties

COMMERCIAL APPLICATIONS

- Microelectronic and nanoelectronic manufacturing for logic, memory, and application-specific (e.g., systems-on-a-chip)
- Emergent metrological tools for R&D
- Chemical and bio-sensors for life sciences, health care, and homeland security

CHALLENGES & BARRIERS

Technical Challenges

- Statistically significant methods that determine nano-property distribution over the chip, across the wafer, and wafer to wafer
- Tools for high-throughput, non-destructive, low cost, high volume manufacturing capabilities
- Tools with relevant sensitivity, resolution, and precision
- Standardized methods and models for analysis of metrology data
- Hierarchical multiscale modeling methods to correlate measured physical properties and device electrical properties

SCOPE OF R&D

Near Term:

- In-line non-destructive critical dimension metrology and instrumentation with 0.3 nm resolution
- Critical metrology for characterizing heterogeneous films and wafers with devices made from these films; e.g., SiGe, strained Si, and SOI
- Critical metrology for vertical films and structures; e.g., barriers, FINFET gates
- Metrology and instrumentation for characterization of metal-high-k gate stacks and other new material systems

Mid Term:

- In-line non-destructive critical dimension metrology instrument with sub-nm resolution

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry, Government – cost-sharing
Industry – technology development
Federal Laboratories – screening, testing, tech transfer (DOE, DOD, NIST); standards, validation (NIST)
Universities – samples and R&D
Industry, Government, Suppliers – reliability (DOE, NIST)
End-User – defines needs

RISK

LOW — HIGH

Technical Risk

May not achieve sufficient sensitivity, resolution or precision

Commercial Risk

May not achieve sufficient speed, low cost

IMPACTS

LOW — HIGH

Extend CMOS lifetime significantly

Enable creation of new nano-enabled chemical and biological sensors and actuators

IMPLEMENTATION STRATEGIES A key implementation strategy is to elicit specific funding for high risk, developmental instrumentation appropriate for large industrial metrology manufacturers that have no access to SBIR. Metrology oriented partnership should be established between universities and government laboratories. Another key strategy is to ensure the accessibility of expensive tools (e.g., more centers of measurement, more support people at the measurement centers).

DEVELOPMENT TIMELINE

2005	2010	2015
CD dimension metrology and instrumentation with 0.3 nm resolution; metrology for heterogeneous and vertical films	CD critical dimension metrology with sub-0.3 nm resolution; metrology for new advanced (beyond) CMOS devices and new instruments	

Priority Topic 4.2. Nanoelectronics Challenge
Instrumentation & Metrology for Emerging Novel Devices & Structures (Beyond CMOS)

VISION AND GOALS In the future, instrumentation and methods will be available for analysis of atomic scale physical, electrical, and chemical properties, as well as methods to correlate these properties with novel, emerging device (beyond CMOS) and system performance.

KEY PERFORMANCE REQUIREMENTS

- Spatially resolved characterization (1, 2 and 3D as appropriate) of physical properties, ranging from nanometer to atomic scale
- Physical properties of interest: chemical composition, electrical, thermal, structural, mechanical and optical properties
- Measurements that provide understanding and control of the nanoelectronic device interfaces
- Robust electrical test structures that provide reliable measurements of nanoelectronic structures, i.e., molecular electronics, carbon nanotubes, semiconductor nanowires

COMMERCIAL APPLICATIONS

- Microelectronic and nanoelectronic manufacturing for logic and memory applications
- Emergent metrological tools for R&D
- Chemical and bio-sensors for life sciences, health care, and homeland security
- Inexpensive, ubiquitous electronics

CHALLENGES & BARRIERS

- Hierarchical multiscale modeling methods to correlate measured physical properties and device electrical properties
- Tools with relevant sensitivity, resolution, and precision
- Standardized methods/models for analysis of metrology data
- Improved contacts and electrical characterization
- Develop reliable, robust test platforms

SCOPE OF R&D

Near Term:

- Develop critical instrumentation, methods, and comprehensive understanding of the key emerging novel device properties
- Identify critical physical/electrical properties of emerging devices
- Develop improved characterization methods and understanding of electrical properties by using prototypical device structures

Mid Term:

- Develop models and simulation methods for metrology tools that give realistic, accurate, and consistent interpretations

Long Term:

- Develop necessary metrology tools for process monitoring that will enable low cost, rapid manufacturing

PARTNERSHIP ROLES AND RESPONSIBILITIES

Industry, Government – cost-sharing (NSF, DOE, NASA)
Industry – technology development
Federal Laboratories – screening, testing, tech transfer (DOE, DoD, NIST); standards, validation (NIST)
Universities – samples and R&D
Industry, Government, Suppliers – reliability (DOE, NIST)

RISK

LOW — HIGH

Technical Risk

May not achieve sufficient sensitivity or resolution

Commercial Risk

May not achieve sufficient speed, low cost; development of metrology platforms for technologies that do not reach commercial viability

IMPACTS

LOW — HIGH

Continuation of Moore's Law beyond physical limitations of CMOS scaling

Creation of fundamentally new nanoelectronic devices

Creation of nano-enabled chemical/ biological sensors and actuators

Enable pervasive electronics (inexpensive nanoelectronic sensors/devices)

IMPLEMENTATION STRATEGIES Key strategies are to elicit specific funding for high risk, new metrologies that target key performance requirements; to initiate metrology-oriented partnerships between industry, university and government laboratories; and to develop centers of research with synergies between advanced nanofabrication and nanocharacterization metrologies.

DEVELOPMENT TIMELINE

2005	2010	2015
Instrumentation and methods; critical physical/electrical properties of emerging devices; characterization via prototyping	Models and simulation methods	Metrology tools for process monitoring for rapid manufacturing

Priority Topic 4.3. Nanophotonics Challenge
Materials and Devices for Nanophotonics

VISION AND GOALS In the future, use of nanophotonic materials and devices will expand in diverse applications such as optoelectronics, data storage, and efficient light sources. The supporting goals are to design, model, and synthesize nanophotonic materials and devices to meet performance requirements at all stages of development.

KEY PERFORMANCE REQUIREMENTS

- Abrupt interfaces with controllable band offsets and confirmed states
- Control of thickness, composition, and uniformity of films and heterostructures
- Functional integration of heterogeneous materials

COMMERCIAL APPLICATIONS

Nanophotonics will serve as an enabling technology in a myriad of applications, including:

- Optoelectronics
- Data storage
- Efficient light sources
- Sensors
- Optical computing
- Biomedical instrumentation

CHALLENGES & BARRIERS

- Controllable synthesis methods
- Interface control in heterostructures
- Integration of heterogeneous materials and devices

SCOPE OF R&D

- Optimization of growth of low dimensional structures and heterogeneous systems
- Coordination between growers, modelers, and metrologists and with instrument manufacturers
- Develop a database for material properties under practical conditions (elevated temperature, strain, processing history, etc.)

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing (NSF, DOE, DOD, DOC, NIH, NASA, DHS)

Industry – cost-shared partnerships between end-users and government

Universities – research collaborations with industry/gov't.

Federal Laboratories – collaborative research within DOE, NIST, DOD, NASA and other agencies

RISK

LOW — HIGH

Technical Risk

Many existing efforts in government, industry and academia lower risks but significant hurdles remain

Commercial Risk

Research in very early stages.

IMPACTS

LOW — HIGH

New biomedical applications

New photodynamic theory

New uses for homeland security

Advanced sensors for bio-agents

Enable secure communications

IMPLEMENTATION STRATEGIES Implementation will be accomplished through partnerships between industry, academia and government.

DEVELOPMENT TIMELINE

2005	2010	2015
Establish effort to promote information exchange among growers, modelers and metrologists	Database of materials properties under practical conditions for nanophotonics developed and in use	Commercial design/modeling packages incorporating full nanophotonics device characterization in use

Priority Topic 4.4. Nanophotonics Challenge
Techniques and Instrumentation for Nanophotonics

VISION AND GOALS In the future, advanced instrumentation and techniques will be developed for applications including nanoscale 3D imaging, chemical analysis, and material manipulation in hard and soft materials. Key goals include 10-fold improvements in instrument performance; enhanced sub-surface imaging and 3D tomography of buried interfaces and biological samples; and integration of nanophotonic devices and materials with different functionalities.

KEY PERFORMANCE REQUIREMENTS

- Accomplish concurrent signal, speed, and resolution
- Subsurface imaging and 3D tomography
- System-level modeling capabilities
- Non-invasive, non-destructive imaging

COMMERCIAL APPLICATIONS

Advanced imaging, chemical analysis, and material manipulation techniques and instrumentation will support advances in the following areas:

- Data storage
- Efficient light sources
- Sensors
- Optical computing
- Biomedical instrumentation

CHALLENGES & BARRIERS

- Integration of devices and material with different functionalities
- Difficulty in acquiring sufficient information to obtain desired properties in sub-surface imaging
- Need new components and devices to improve signal

SCOPE OF R&D

- Instrumentation and metrology to support component- and system-level integration of heterogeneous nanophotonic devices and materials
- Development of enhanced sub-surface imaging for buried interfaces, *in vivo* biological samples
- Evolutionary and revolutionary improvements in signal, resolution and speed

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing (NSF, DOE, DOD, DOC, NIH, NASA, DHS)

Industry – end-user led R&D

Universities – research collaborations with industry/gov't.

Federal Laboratories – collaborative research with DOE, NIST, DOD, NASA and other agencies

RISK

LOW — HIGH

Technical Risk

Many difficult problems, sub-surface imaging and spectroscopy and biological systems particularly challenging

Commercial Risk

Instrumentation must also be cost effective for widespread use

IMPACTS

LOW — HIGH

Sub-cellular imaging of process' life

Communications and data storage

In-situ metrology

Characterization of new materials & devices

New modeling algorithms

IMPLEMENTATION STRATEGIES Implementation will be accomplished through partnerships between industry and government to leverage technical risk.

DEVELOPMENT TIMELINE

2005	2010	2015
Initiate campaign to provide 10x improvement in signal-to-noise, resolution, speed	Sub-surface spectroscopy in 8 hours	Spectroscopy at nanometer scale ($1nm^3$) in 1 hour

Priority Topic 4.5. Nanomagnetics Challenge
Measurement of Magnetic Properties of a Cubic Nanometer of Material

VISION AND GOALS In the future, instrumentation and metrology will be available to measure and analyze the magnetism in the small amounts of material that will be incorporated into magnetic and spin-electronic devices. It will also be possible to probe smaller regions than ever before, thereby leading to the discovery and utilization of novel magnetic phenomena for new device paradigms.

KEY PERFORMANCE REQUIREMENTS

- Measure a magnetization of 10^{-20} A·m^2 (10^{-17} EMU) =1000 Fe atoms
- Measure interparticle and intergrain interaction (*in situ*) with spatial resolution less than 1 nm^3
- Ability to probe buried interfaces in layered structures
- Ability to deduce bulk property values from local measurements
- Detetermine magnetic polarization of 1 nm^3

COMMERCIAL APPLICATIONS

- High saturation M permanent magnets
- Devices for controlling biological action in cells
- MRAM
- Hard disk with 1.5 Tb/cm^2 (10 Tb/in^2) storage capacity
- Low loss transformers and generators

CHALLENGES & BARRIERS

- Ability to manipulate samples at the nanoscale
- Lack of magnetic sensitivity
- Lack of spatial resolution
- Theory for measurement analysis in order to bridge magnetic theory with phenomena
- Measurement accessibility (cost, size, portability of appropriate measurement techniques)
- Lack of appropriate samples for measurement; in situ measurement capabilities

SCOPE OF R&D

- Develop key instrumentation and understanding of new device properties
- Develop improved characterization methods and understanding of magnetic properties by using prototypical device structures
- Develop models and simulation methods for metrology tools that give realistic, accurate, and consistent interpretation
- Develop necessary metrology tools for process monitoring that will enable low cost, rapid manufacturing

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing and validation

Industry – cost-sharing, development

Federal Laboratories – screening, development, testing, variables, and standards

Universities – samples, development, validation

Supplier – reliability

RISK

LOW — HIGH

Technical Risk

May not achieve sufficient sensitivity or resolution

Commercial Risk

Particle beams for probing samples are getting smaller, but sensors for detecting the smaller numbers of scattered particles may not be developed

In situ industrial measurement techniques may not be developed

IMPACTS

LOW — HIGH

Increase magnetic recording density to 1.5 Tb/cm^2 (10 Tb/in^2)

Improve MRI resolution by 10^3

Inexpensive, non-volatile MRAM, more reliable than FLASH memory

Permanent magnets with saturation magnetization greater than 2T

Magnetic control of biological processes in cells

IMPLEMENTATION STRATEGIES Develop metrology-oriented university/industry/government partnerships; develop centers of research on measurement science; incorporate new tools into industrial production lines.

DEVELOPMENT TIMELINE

2005	2010	2015
New field and beam instrumentation and metrology concepts	New generation of fast high sensitivity field and beam sensors; New models and simulation methods	Incorporate new sensors into tools for process monitoring and rapid manufacturing

Priority Topic 4.6. Nanomagnetics Challenge
Imaging of Spin Dynamics

VISION AND GOALS In the future, imaging tools at the nanoscale that are accurate, reliable, cost-effective, and suitable for applications ranging from basic research to commercial products will be necessary for advancement of nanomagnetics. The development of instrumentation for imaging spin dynamics at the nanoscale with deep sub-nanoscale time will support this vision.

KEY PERFORMANCE REQUIREMENTS

- 3D magnetization measurement
- Less than 10 nm spatial resolution
- Deep sub-nanoscale resolution in real time (not averaged over many cycles)
- Element specificity is highly desired

COMMERCIAL APPLICATIONS

Imaging and spin dynamics nanotechnology will find uses in several applications, including:

- Data storage
- Disk drives
- MRAM (magnetic random access memory)
- Sensors (bio-health, homeland security, MEMS)

CHALLENGES & BARRIERS

- Ability to manipulate samples on nanoscale
- Accurate modeling of detection probe—analytical and micromagnetic theory
- Fast and large bandwidth electronics for fast data acquisition
- Delivery of fast, spatially localized, high-field pulses
- High sensitivity detectors; scanning probe response time
- X-ray optics

SCOPE OF R&D

- Develop fast, non-destructive measurement probes
- Develop tools to quickly manipulate or deliver sample to the sensor or experiment
- Develop tools to measure samples under normal operating conditions
- Improve time and spatial resolution of x-ray and other imaging tools

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing and validation

Industry – cost-sharing, development

Federal Laboratories – screening, development, testing, variables, and standards

Universities – samples, development, validation

Supplier – reliability

RISK

LOW — HIGH

Technical Risk

May not achieve sufficient speed, sensitivity, or resolution

Commercial Risk

May not achieve *in-situ* measurement

IMPACTS

LOW — HIGH

Enable tests of nanoscale theory
Creation of Read and Write devices for nanoscale storage media

Creation of spin-dependent electronics and detection systems, magnetic imaging of biological activity, smaller motors and generators, more efficient transformers

IMPLEMENTATION STRATEGIES Develop metrology-oriented university/industry/government partnerships; develop centers of research on measurement science; incorporate new tools into industrial production lines.

DEVELOPMENT TIMELINE

2005	2010	2015
Demonstrate localized high field pulse source. Select probes (photons, x-rays, neutrons, electrons)	Demonstrate sub-nanoscale imaging resolution with appropriate time scale	Commercialize tools, standards

Priority Topic 4.7. Nanomagnetics Challenge
Measurement of Spin Transport in Materials

VISION AND GOALS Development of the appropriate tools for measuring spin current in semiconductors and other materials is critical to the advancement of spin electronics (spintronics). To achieve this goal, researchers will begin by developing a fundamental understanding of the spin current theories and draw upon this knowledge to develop the required metrology tools and techniques.

KEY PERFORMANCE REQUIREMENTS

- Ability to measure spin polarization and its current directly
- Ability to deduce spin current from measurements of spin accumulation
- Ability to measure single tunnel barriers

COMMERCIAL APPLICATIONS

The ability to measure spin directly will find use in several applications, including:

- Spin transistors
- MRAM
- Quantum computers

CHALLENGES & BARRIERS

- Lack of ferromagnetic semiconductors with Curie temperatures well above 300 K
- Probes that decouple the measurement from the phenomenon
- Method for measuring a single electron spin – only way of getting it now is via the creation of a photon and measuring that photon
- Lack of a generator of a single electron spin
- Probe for measuring magnetoresistance locally

SCOPE OF R&D

- Develop a method for injecting spins into a semiconductor without changing their spin direction
- Develop probe to measure a spin *in situ* in a way that it is distinct from its charge
- Improve modeling capabilities to be able to infer spin polarization current from spin accumulation
- Develop capability to measure spins in Si
- Develop an efficient spin transistor

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-sharing and validation

Industry – cost-sharing, development, and validation

Federal Laboratories – screening, development, testing, variables, and standards

Universities – samples, development, validation, modeling

Supplier – reliability

RISK

LOW — HIGH

Technical Risk

"High risk, many uncertainties"; may not succeed in injecting and controlling an electron spin in a semiconductor

Commercial Risk

High risk, but big payoff; materials and structures may not be developed to implement the predictions

IMPACTS

LOW — HIGH

Would revolutionize the electronics industry as devices would be created on the basis of the spin density of states of a material rather than its charge density of states; Could lead to quantum computation

Opens venues for new device designs, like faster electronic switches and remotely actuated switches

IMPLEMENTATION STRATEGIES Develop metrology-oriented university/industry/government partnerships; develop centers of research on measurement science; incorporate new tools into industrial production lines.

DEVELOPMENT TIMELINE

2005	2010	2015
Demonstrate new spin detection schemes; improve tunneling barrier processing	Improve modeling to be able to infer spin current from spin accumulation data, and to bridge between different size scales	Develop probe to measure spin directly and separately from charge

area, most equipment is being used at the current limits of resolution, and new equipment is needed to probe even smaller volumes and with lower uncertainties.

Along with new instrumentation, a central database of nanoscale information is needed to serve as a resource for standardized reference materials for nanometrology. Greater knowledge of future processes, materials, and structures will be needed to facilitate and target R&D for new metrology. An infrastructure that incorporates knowledge, expertise and technology capabilities must be developed to promote the development and validation of new metrology methods for emerging devices and systems.

Probing nanoscale devices and systems will require revolutionary developments rather than evolutionary advances in measurement schemes and devices. However, incentives are currently lacking to encourage scientists in academia to focus on instrument research and development. In addition, the large equipment manufacturers who would be interested in development of new equipment are precluded from some of the funding opportunities that could leverage the high cost of instrumentation development (e.g., SBIR). Another issue is that the current workforce is lacking both an understanding of nanoelectronics, nanophotonics, and nanomagnetics phenomena and an understanding of measurement science needed to develop new tools.

IMPLEMENTATION STRATEGIES

Strategies for implementation of a concerted effort to accelerate development of required nanoinstrumentation and metrology in nanoelectronics, nanophotonics and nanomagnetics include development of nanotechnology measurement centers, integration of R&D resources and training, and funding opportunities as described below.

Consolidation of resources into centralized nanotechnology centers is needed to provide greater accessibility to expensive high-technology metrology techniques. Resources could be pooled in technology centers that offer expertise in specialized fields and technology capabilities such as newly developed instrumentation or test bed fabrication. Such centers would require significant staffing to assist outside users in both measurement and analysis of the data and ensure effective use of their tools. Another strategy is to establish university centers to focus on new approaches to basic R&D in electronic, photonic, and magnetic metrology. An important strategy will be to promote integration from the supplier to the application, in terms of equipment, education and training, and device or system.

Funding should be provided for interdisciplinary research groups focusing on instrumentation needs. One approach is for different funding agencies to create separate, dedicated funding sources for supporting the development of new measurement tools. This focused funding in metrology could be used to encourage universities to include measurement tool development as a criterion for promotions and awarding of tenure. Another incentive to increase activity in instrumentation and metrology could be to create an annual award related to nanoscale tool development (e.g., Nanotool of the Year).

SUMMARY

Electronics, photonics and magnetics are pervasive fields of science and technology with large effects on many industrial sectors, including electronics, computing, health care, biotechnology, energy, transportation, homeland security, telecommunications, nanotechnology, sensors, and defense. In the future, new nanoscale devices and structures are expected to revolutionize the fields of nanoelectronics, nanophotonics, and nanomagnetics. As the size of the device is reduced to the

nanoscale, the volumes of the electronic, photonic, and magnetic elements are also decreased, leading to significantly reduced voltages, luminosity, and magnetization values. Greater sensitivity in measuring devices will be needed to measure the properties of these systems. In addition, because the physics of the nanoscale can be significantly different from that of large-size structures, it is not at all obvious that present-day metrology will be applicable in the nanoworld. Realizing the effect of these imminent advances in the nanoelectronic, nanophotonic, and nanomagnetic fields will consequently require an accelerated development of the underlying metrology and instrumentation needed to make reliable, reproducible measurements of device performance and materials' properties and to successfully incorporate devices into commercial products.

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5. Instrumentation and Metrology for Nanofabrication

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SCOPE

Nanofabrication involves methods of fabricating structures that are generally part of a functional device or play a role in contacting the device. These structures may have features with lateral dimensions as small as a single atom. Much of this work is currently focused on fabricating features that are not necessarily accessible to the macroscopic world. Techniques such as modification of hydrogen-terminated surfaces and directed alteration of self-assembled monolayers are being widely explored, as they are often stable in an ambient environment. There is also a significant focus on the individual manipulation and placement of atoms and molecules. Although much of the atomic-scale manipulation takes place in an ultra-high vacuum environment, future work will involve developing methods to interact with these structures and devices with external instrumentation.

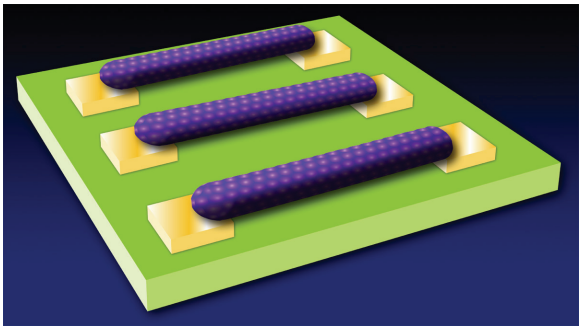


Figure 5.1. Schematic representation showing possible future devices and fabrication techniques (nanotubes on contact pads). With the appropriate electronic structure these can possibly be functional devices (courtesy of NIST).

The nanofabrication section is focused on the broad question of which technologies are most promising, where additional research should be focused, and identifying key challenges in the transition from research and development to a manufacturable technology. Other key issues such as interconnectivity are also recognized as essential and integral to the future of nanofabrication.

Vision for Nanofabrication

Nanofabrication will be the infrastructure that enables revolutionary solutions (assembly from atomic/molecular constituents), including tools to enable top-down, high-volume solutions. Nanofabrication will include the:

- *Ability to fabricate, by directed or self-assembly methods, functional structures or devices at the atomic or molecular level*
- *Ability to probe individual nano devices by either scaled contact methods or more sophisticated optical/electrical non-contact approaches*

VISION

Nanofabrication is the ability to fabricate, by directed or self-assembly methods, functional structures or devices at the atomic or molecular level. By virtue of its microscopic scale, nanofabrication often leads to bottom-up or locally ordered solutions in applications where top-down or globally ordered organization is not required or feasible. Over time, successful high-volume implementation of bottom-up technologies will allow functional elements fabricated using the bottom-up approach to be used in top-down applications, as the capabilities of nanofabrication tools mature.

For example, a bottom-up application of nanofabrication would be a tool that could manufacture nanofabricated quantum dots in bulk form, such that they could be used as an additive to a bulk material (such as adding them to an optical element to use an optical property of the quantum dots). A subsequent top-down application of such a nanofabrication system would be to devise a further nanofabrication methodology that would place these quantum dots in desired locations such that they could be incorporated into an ordered system (such as a monolithic optoelectronic circuit).

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

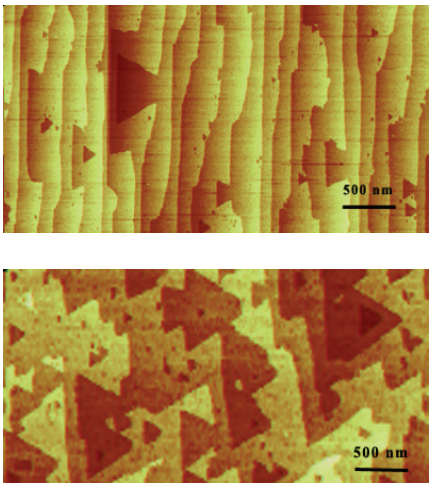


Figure 5.2. An example of an atomically ordered hydrogen-terminated silicon surface produced at NIST. These substrates can be subsequently used as catalysts for directed assembly and modified self-assembled structures (courtesy of NIST).

The broad scope of existing and emerging techniques for nanofabrication is shown in Table 5.1. There are a number of excellent examples of *directed atomic or molecular manipulation using scanning probes*, demonstrating manipulation of atoms and the creation of new structures with specific nanometer scale functionality. Examples include electron corrals and other quantum-type systems in which the arrangement and manipulation of individual atoms forms a unique structure that depends directly on the atomic arrangement. The main challenge in this class of fabrication is that it is viewed as a serial methodology and is not feasible for large-scale manufacture, although it does provide a useful research foundation.

Directed self-assembly (involving guidance and capture) is another key method. This is a methodology in which self-assembly-type methods are applied except that the self-assembly has a directed or ordered aspect. Self-assembly of molecules typically occurs in a layer-by-layer growth process and elements of the growth process cause one layer to organize in a specific manner relative to another layer. Forces can be applied and, for example, molecules can "screw" into one another or assemble in complex 3D ways.

Array technology is essential to advancement of scanned probe methods as used in atom assembly. Some results of up to a 1000 × 1000 probe array have been demonstrated. For scanned probe methods to become a viable manufacturing alternative, substantial advance is needed in probe arraying techniques well beyond the limited work and current focus.

Table 5.1
Existing and Emerging Nanofabrication Methodologies

• Direct atom molecular manipulation with scanning probe • Array technology (up to 1000 × 1000) • Beam technology (ebeam, serial, parallel) • Directed self-assembly (involving guidance and capture) • Templating (e.g., molecular, surface, etc.) • Film deposition methods • Biological/bioassembly techniques (all self-directed assembly) • Structured light/optical lattices/atom optics • Continuous flow systems (microfluidic)	• Nanoimprint • *In situ* analytic tools • Light-based lithography • Laser tweezer • Atomic ink jet • Micro tweezers • Mixed mode lithography • Decoration + super selective trap • Controlled surface reactivity • Nanopositioning (repeatable, accurate, linear) • Self-aligning metrology

Templating (e.g., molecular, surface) is another critical class of fabrication methods. In this arena some substrate or covering layer is modified. An example is hydrogen-terminated silicon surfaces. In this case, the hydrogen termination is broken, and an oxide forms as a hard etch mask. Patterns at or near to the atomic scale can then be formed and transferred into the underlying substrates. Templating methods have been documented with several laboratory examples. The key challenge is a lack of massive parallelism.

Biological/bioassembly techniques (all self-directed assembly) represent another set of methods that have significant potential. Bio-assembly involves more complex mechanisms than simple physical forces and constraints. Biological/chemical reactions can be harnessed to drive complex assembly in both two and three dimensions.

Table 5.2
Challenges and Gaps in Nanofabrication Technology

Category	Technology Gap
Manufacturing, Implementation, and Manufacturability	• Ultra high accuracy 3D positioning (picometer) over large volume (1 cm^3) or length scale • Methods to attach single molecules on surfaces with specific orientation and density • Control of 3D synthesis of nanostructures • Higher throughput metrology capable of near-atomic resolution • Nanoscale test sites as reporters/functionality beacons • Global navigation to a nanofabrication structure • Methods of controlling large arrays of tips • Nanostructures (passivation) • Software for design, control, modeling • Batch fabrication of nanoparticles with a low degree of poly-dispersity • Precise circular dichroism standards at the nanometer scale • Environmental impact management
Nanometer Scale Science and Technology	• Lack of atomically precise tips for AFM, STM • Monitoring dynamics of assembly • Surface science for biological materials • Theory, modeling, simulation to support nanofabrication and metrology for nanofabrication • Component interconnectivity • Bridging from the microscale to the nanoscale • Inability to etch/sculpt features on a nanometer scale • Interfacing atomic scale devices • Link/bridge from inorganic material to biological material • Simulation and physics-based models to interpret metrology data • Intrinsic atomic length scales (self-referencing) • Detection of fluorescence signal at a single photon level and conversion to image • Noncontact inspection of device performance • Noncontact standing methods (electron, X-ray) for wireless contact to nanodevices

GOALS, BARRIERS, AND SOLUTIONS

A summary of the current barriers and gaps in nanofabrication technology is shown in Table 5.2. The priority associated with each of these challenges is illustrated by the symbols that appear next to each idea (priorities voted on by workshop participants). As shown in Table 5.2, critical gaps exist in manufacturing and manufacturability of nanofabricated systems, and in the enabling nanometer scale science and technology that will be required for effective nanofabrication methods.

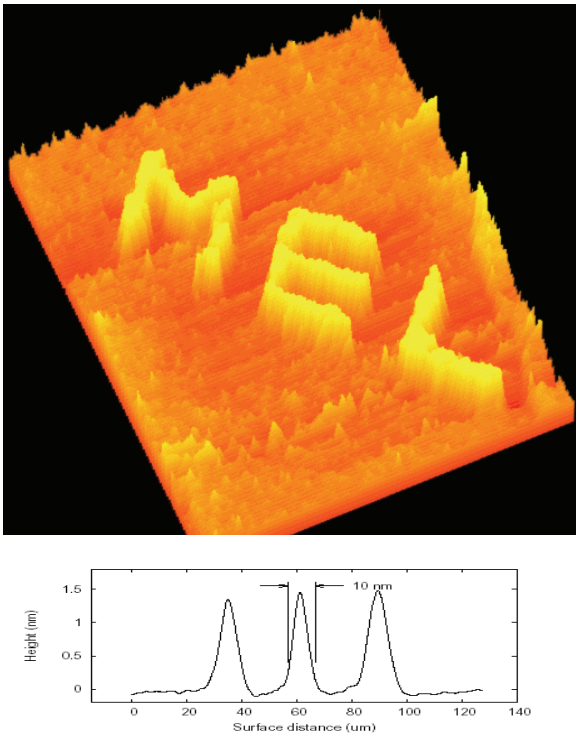


Figure 5.3. This figure shows features written with an STM nominally 10 nm in size. These features and patterns can be transferred into the silicon substrates for future processing and device applications (courtesy of NIST; reprinted with permission from [1]).

A key goal is to develop the technology to effectively enable the transition from the microscale to the nanoscale and ultimately the atomic scale. Typically referred to as the interconnectivity problem, it is widely recognized that an enormous challenge exists in making directed contact to the atomic- or nanometer-scale world. Significant barriers arise in attempting to contact the dense nanometer-scale environment with the macroscopic world, as well as when interfacing atomic-scale devices (i.e., electrical, chemical).

The lack of *large-scale accurate positioning systems* is a substantial problem, as these must be used extensively in the fabrication and metrology of nanofabricated structures. Systems are required to enable global navigation to the nanofabricated structure. Ultra-high accuracy 3D positioning (at the picometer scale) over large volumes (1 cubic centimeter) or length scales is needed. The goal is to achieve accurate subnanometer-resolution-stage positioning that couples an area hundreds of millimeters in size with accurate nanometer positioning.

The growth and assembly of complex 3D structures represents a much more complex challenge than simple 2D structures and growth processes. The ability to measure and control nanostructure synthesis processes in three dimensions in real time will be a critical hardware challenge for future nanofabrication technology and is an important goal.

Modeling and theoretical simulation are essential elements of fabrication at the nanoscale. Models need to be developed that accurately represent the nanometer scale and support nanofabrication as well as metrology for nanofabrication. Atomic forces and other criteria that cause assembly to occur need to be fully modeled to move these methods into accurate manufacturable technologies with reliable manufacturing processes.

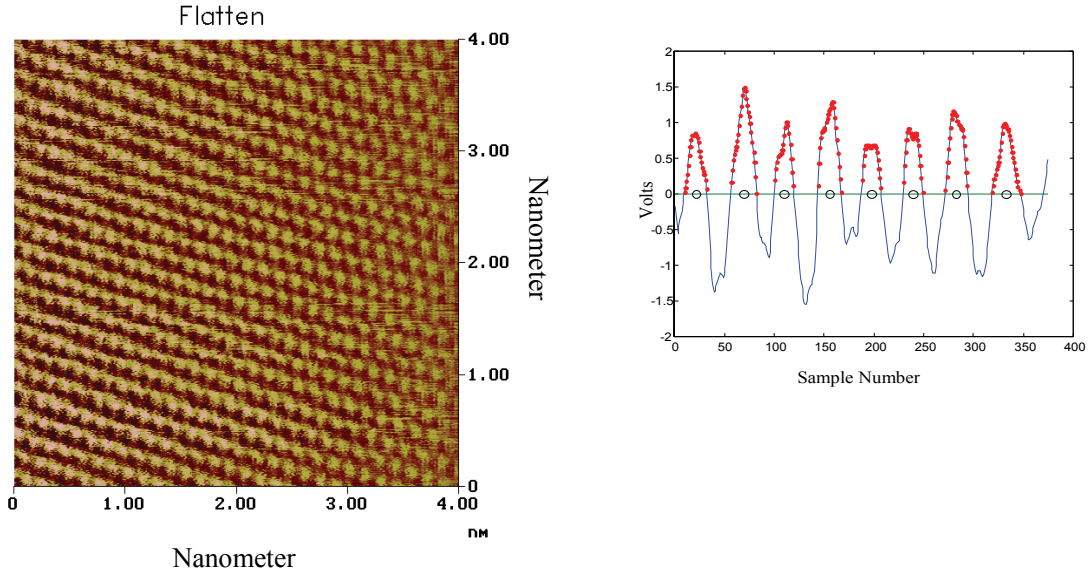


Figure 5.4. Shown is an atomic resolution STM scan of a graphite lattice. The right panel is an interferometer measurement showing picometer resolution data. Each peak is associated with an atomic lattice site. This method was developed to measure positions and placement to well below a nanometer (reprinted with permission from [2]).

Another critical goal is to overcome the challenges associated with the massive assembly of atomic scale structures using direct atomic manipulation. For single atom directed assembly methods to advance or become suitable for large-scale manufacturing, significant progress must be made in parallelism. Complex assembly hardware control code running thousands or millions of assemblers in parallel needs to be developed in concert with large tip arrays that operate independently.

R&D INVESTMENT AND PRIORITY RESEARCH AREAS

Based on the existing goals, barriers that must be overcome, and gaps in technology, five priority challenge topics have been defined for nanofabrication. These topics are outlined below in order of priority and are discussed in more detail in Priority Topics 5.1–5.5.

- *Interconnectivity between the macroscopic and atomic-length scales:* nanofabrication involves fabricating device-like structures with features having dimensions as small as a single atom. The ability to make direct contact between the macroscopic (i.e., external instrumentation) and the atomic- or nano-scale world will be key to successful nanofabrication techniques and is a critical R&D path.
- *High-accuracy 3D positioning:* technology is needed to accurately position (picometer scale) a nanoparticle or structure over large volumes (1 cm^3) or lengths and will be critical to the fabrication of nanostructures and devices. Accurate positioning will also enable navigation to the nanofabricated structure.
- *Control of 3D synthesis of nanostructures:* technology is needed to effectively control the growth and assembly of complex 3D structures. Hardware challenges in measurement and control need to be addressed.

- *Theory, modeling, and simulation in support of nanofabrication:* models must be developed to accurately represent the nanometer scale and support associated nanofabrication metrology. These modeling tools should be robust enough to enable reliable manufacturing processes.
- *Assembling atomic-scale structures with direct atomic manipulation:* technology is needed to overcome the challenges of massively assembling atomic-scale structures with direct atomic manipulation, particularly with respect to parallelism. Priorities include assembly hardware control code with the capability to run thousands or millions of assemblers in parallel, coupled with atomistically precise large tip arrays.

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

The technical infrastructure to support nanofabrication needs to be expanded to include local and regional research centers (larger facilities allowing access to shared cost instrumentation and resources), as well as access to selected Federal research centers (e.g., national laboratories, NIST). Coordination of research at private and public research centers should be undertaken with industrial guidance along the way. Consideration should be taken to evaluate duplication of research efforts at the national level.

Education and training opportunities should be identified and pursued. Such activities will help to build the underlying scientific basis for this important field and support creation of new technology and products in the future.

Funding should be leveraged through Federal and private cost-sharing as appropriate. One approach is to create special SBIR topics that would support basic nanotechnology studies with an application-specific focus. Funding could be targeted for small, niche application development.

IMPLEMENTATION STRATEGIES

Suggested implementation strategies for nanofabrication include the following:

- A continuing set of workshops to focus Federal R&D funding on critical areas of research.
- Specific funding targeted for basic research at the university level. It is a strong opinion that creative, smaller research efforts not tightly controlled are necessary at this stage.
- Development of university, government, industry collaborations as “centers of excellence.” This is essential to allow collaboration among researchers with the various types of expertise required. Also, some very high-dollar fabrication capabilities such as ebeam writers are best operated in a shared manner. The center for excellence concept is intended to make expensive resources and expertise available more broadly with a shared cost basis.
- Supporting and encouraging the required overlap in disciplines. R&D in nanotechnology requires strong interdisciplinary groups. To a greater extent than virtually any other emerging field, the multidisciplinary requirements for materials scientists, chemists, physical chemists, physicists, electrical engineers, mathematicians and modeling experts working closely together must be strongly supported.
- Ensuring that personnel requirements for the future are met by appropriate training and graduating new personnel with the needed skill sets.
- A structured set of workshops with outputs intended to focus and direct resources beyond Federal R&D funding. Specific gaps should be identified in both funding and effective research.

Priority Topic 5.1. Nanofabrication Grand Challenge
Interconnectivity of Macroscopic and Atomic Length Scales

VISION AND GOALS Nanofabrication involves fabricating structures which have dimensions as small as a single atom and either display device characteristics or are a part of making contact to nanometer scale devices. The ability to make direct contact between the macroscopic (i.e. external instrumentation) and the atomic- or nano-scale world will be key to successful nanofabrication techniques.

KEY PERFORMANCE REQUIREMENTS

- Ability to contact a dense nanometer-scale environment with the macroscopic world (i.e. external instrumentation)
- Interfacing atomic-scale devices (i.e. electrical, chemical)

COMMERCIAL APPLICATIONS

- Arrayed nanometer scale devices (will require substantial interconnect architecture)
- Well-controlled electronic and mechanical interfaces (most devices)
- Contact and interfacial boundaries in computation, memory and MEMS structures
- Semiconductor manufacturing: contacts, vias, etc

CHALLENGES & BARRIERS

- Non-contact inspection of device performance
- Non-contact standing wave methods (electron, x-ray) for wireless connection to nanodevices
- Interfacing atomic scale devices to nanodevices and nanostructures

SCOPE OF R&D

- Local probe measurements of interfacial electronic structures
- University and industrial research on non-conventional strategies for interconnects
- Arrayed methods for large numbers of individual data exchange
- Understanding probe and contact interations and effects on devices and device performance

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D
Industry – cost-shared partnerships with government

Federal Laboratories – screening and testing

Universities – experimental validation.

Suppliers – cooperate in R&D with federal laboratories

End-Users – participate in activities

RISK

LOW — HIGH

Technical Risk

Commercial Risk

IMPACTS

LOW — HIGH

Understanding of interfaces

Flexibility

Relevance to commercial applications

Reliability

Economies of use (system-dependent)

Priority Topic 5.2. Nanofabrication Grand Challenge
High Accuracy 3D Positioning Over Large Volumes or Length Scales

VISION AND GOALS The ability to accurately position (picometer scale) a nanoparticle or structure over large volumes (1 cm^3) or lengths is critical to the fabrication of nanostructures and devices. Accurate positioning will enable effective navigation to the nanofabricated structure.

KEY PERFORMANCE REQUIREMENTS

- Positioning that couples 100s of mm range with nanometer accuracy
- Ability for global navigation to a nanofabricated structure
- Accurate and repeatable nanopositioning technologies

COMMERCIAL APPLICATIONS

- Lithography processes at the nanometer scale
- Accurately sampling targets of interest (probes)
- Level to level positioning, overlaying atomic level process steps
- Accurate alignment of contacts and vias to lower level structures

CHALLENGES & BARRIERS

- Lack of global navigation to nanofabrication structures (i.e., nanoscale GPS)
- Ability to accurately position nanostructures over a large volume (1 cm^3) or length scale
- Intrinsic atomic length scales (self-reference)

SCOPE OF R&D

- Interferometry with picometer accuracy
- New positioning systems that are sensitive to local environment
- Stage positioning hardware improvements
- Tip positioning with global vision system interlock and control
- Closed loop feedback systems with sub-nanometer accuracy

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – collaborative research with industry and federal laboratories

RISK

LOW — **HIGH**

Technical Risk

Commercial Risk

Priority Topic 5.3. Nanofabrication Grand Challenge
Control of Three-Dimensional Synthesis of Nanostructures

VISION AND GOALS In the future, technology will have the capability for effectively controlling the growth and assembly of complex 3-dimensional structures. Primary challenges exist in development of hardware for measurement and control.

KEY PERFORMANCE REQUIREMENTS

- Ability to measure and control synthesis in three dimensions
- High throughput capability with near atomic resolution

COMMERCIAL APPLICATIONS

- Computation and memory devices using 3-dimensional directed assembly structures
- Optical and photonic structures using complex 3-dimensional chemistry
- Underlying surfaces based on self assembly techniques
- Complex 3-dimensional resist and lithography process
- Interfacial layers in 3D structures

CHALLENGES & BARRIERS

- Ability to measure local atomic characteristics
- Need to sample inside structures with 3-dimensional accessibility
- Making measurements without perturbing the nano-system

SCOPE OF R&D

- Significant chemistry component
- Developing ability to measure local atomic characteristics over large areas
- Non-invasive measurement methods to accurately sample chemical structure
- 3-dimensional imaging methods to reconstruct complete sample structure

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – collaborative research with industry and federal laboratories

RISK

LOW — HIGH

Technical Risk

Commercial Risk

Priority Topic 5.4. Nanofabrication Grand Challenge
Theory, Modeling, and Simulation in Support of Nanofabrication

VISION AND GOALS Modeling and theoretical simulation is an essential element of fabrication at the nanoscale. Atomic forces and other criteria that cause assembly to occur need to be fully understood and modeled. This will enable the development and deployment of accurate nanotechnologies with reliable manufacturing processes.

KEY PERFORMANCE REQUIREMENTS

- Understanding of forces (i.e. atomic force) and phenomena that play an important role in nanofabrication
- Models that accurately represent the nanometer scale

COMMERCIAL APPLICATIONS

Theory and computational methods will support all applications where nanofabrication has an impact (see Figures 5.1-5.3)

CHALLENGES & BARRIERS

- Lack of software for design, control, and modeling
- Lack of simulation and physics-based models to interpret metrology data

SCOPE OF R&D

- Modeling and simulation combined with well-defined experiments to determine material properties at the nanoscale
- Accurate physical theories at the nanometer scale
- Reduced-order or low-order models
- System-level models and descriptions that integrate nanomanufacturing components, sensors, actuators, and control units
- Real-time data analysis and visualization of data

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – collaborative research with industry and federal laboratories

RISK

LOW — HIGH

Technical Risk

Commercial Risk

Priority Topic 5.5. Nanofabrication Grand Challenge
Assembling Atomic Scale Structures with Direct Atomic Manipulation

VISION AND GOALS In the future, single atom, direct assembly methods will enable the assembly of massive atomic-scale structures. Significant progress in parallelism will be necessary for direct atomic manipulation fabrication technologies to advance to the level of manufacturability required for mass production.

KEY PERFORMANCE REQUIREMENTS

- Complex assembly hardware control code to run thousands or millions of assemblers in parallel
- Independently operating large tip arrays (1000 by 1000 and larger)
- Ability to use array technology with a wide variety of materials

COMMERCIAL APPLICATIONS

Direct atomic manipulation technologies that incorporate parallellism will find use in several nanofabrication techniques, including:

- Templating
- Direct atomic manipulation with a scanning probe
- Array technology

CHALLENGES & BARRIERS

- Nanofabrication using direct atom/molecule manipulation with a scanning probe is not feasible for manufacturing scale
- Work in array technology is limited; scanning probe methods require significant technology advancements
- Use of array technology is not applicable to a wide variety of materials
- Improved scanning probe position accuracy
- Lack of atomically precise tips for atomic force microscopy and scanning tunneling microscopy

SCOPE OF R&D

- Development of tools such as laser and micro tweezers
- Increased scanning probe position accuracy
- New scanning probes methods

PARTNERSHIP ROLES AND RESPONSIBILITIES

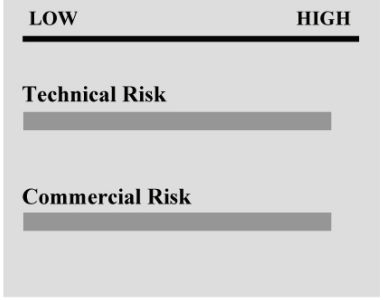
Government – cost-shared R&D (SBIR), targeted funding for small application development

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – provide educational/training opportunities

RISK



- Increased support for modeling and improving the fundamental understanding of physics-based modeling and simulation; technical conferences and focused workshops ensuring the exchange of information for the evolution of improved physics-based models and developing strong agreement between theory and experimental results.

SUMMARY

There are two basic directions identified for the successful development of instrumentation and metrology for nanofabrication: the "bottom-up" approach and the "top-down" approach. The metrology and instrumentation challenges are quite different in some aspects between the two, with other areas of strong parallel requirements. The ability to manipulate, fabricate, organize, and assemble structures on the atomic scale is to be contrasted with fabricating smaller and smaller structures using large lithography-type solutions that essentially scale down fabrication processes. The bottom-up approach will likely involve methods of directed self-assembly, atomic manipulation, and modification of self-assembled substrates, whereas the top-down approach capitalizes on, for example, electron beam and extreme ultraviolet lithography methods for fabrication. The metrology requirements in each case are quite different.

To measure and perform effective process control of individual atomic-scale processes requires a resolution on the atomic scale with the appropriate sampling and statistical models to ensure an accurate measurement of that fabrication element. Measurement methods are needed that effectively sample the actual chemical bonding process or local electronic structure and electronic interactions and provide acceptable feedback to the fabrication instrumentation and hardware. This needs to be accomplished rapidly and with acceptable cost of ownership to enable a profitable process. There are significant challenges that reside in optimizing the atomic-scale imaging at speeds high enough to provide the required process control information. Likewise, the trade-offs between resolution and detailed information content need to be reconciled with high-speed fabrication and profitable nanomanufacturing.

The requirements for top-down approaches must focus on accurate positioning and overlaying of different process levels. Fabrication tools must have very accurate 3D positioning capabilities. The requirements at the atomic scale are well beyond current hardware capabilities and may require interferometry and positioning systems with rapid subnanometer accuracy. In these applications, chemical homogeneity and resist molecule sizes may be limiting factors. An example is that an ebeam writer capable of fabricating 5 nm critical dimension features must have a homogeneous electron beam with resist molecules small enough to not limit feature size. These types of nanomanufacturing tools will no doubt be required at some process stages to enable interconnects and connections to the macroscopic environment.

It is also important to recognize the importance of modeling and the fundamental requirements on physics-based modeling and simulation. At these scales, local atomic forces and interactions are an integral part of the fabrication and metrology process. To understand these behaviors accurately, a thorough understanding of the physics and chemical interactions is necessary.

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6. INSTRUMENTATION AND METROLOGY FOR NANOMANUFACTURING

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SCOPE

Reliable, reproducible nanomanufacturing is critical to the commercial success of future nanotechnology-based products and devices, and instrumentation and metrology are essential components. As nanotechnology transitions from scientific concept to manufacturing, metrology needs change accordingly. The nanomanufacturing industry will need new metrology tools to meet the unique challenges of a nanoscale production environment and ensure that manufacturers can make the measurements that are critical for product and process conformance. Metrology must be brought onto the production floor, where issues such as product throughput, process control, and safety are critical. The scope of this chapter related to nanomanufacturing involves those technologies that have the potential to meet the unique metrology needs for commercial-scale production of nanoscale elements, features, structures, and devices. Currently the semiconductor industry is one of the major nanomanufacturing sectors, hence a good deal of instrumentation and metrology has been developed for that industry. To some extent this suite of instrumentation will be applicable to other forms of nanomanufacturing as they develop. Some evolution of instrumentation and metrology will be needed as new applications are developed, and some revolutionary new instrumentation may be needed.

Advances in metrology for nanomanufacturing will provide a solid foundation for the nanomanufacturing enterprise. The ability to measure, control, and predict the nanoscale structure, performance, and properties of materials and devices over millimeter scales represents a critical enabling technology for nanomanufacturing and is a key focus for research and development.

VISION

The vision for metrology and instrumentation for nanomanufacturing is a tiered infrastructure wherein specialized in-line metrology tools for rapid and precise measurement for process control during manufacturing are backed up by slower, yet more accurate and general, tools off the manufacturing floor or in research laboratories. The highly diverse nanomanufacturing applications of the future will require a similarly diverse set of metrology tools and infrastructure suitable for both low- and high-volume markets.

Vision for Nanomanufacturing Instrumentation & Metrology

Nanomanufacturing in the future will rely on fast in-line metrology tools for process control, backed up by slower, more accurate tools off the manufacturing floor. Tools will be cost-effective, fast, suitable for mass production, occupy minimal floor space, not require ultra-high vacuum or stringent vibration isolation, and support appropriate work volumes. Real-time data will provide fast analysis and control of manufacturing processes.

First-generation metrology tools will include most of the current technologies such as near-field optics, scanning microscopy, spectroscopy, and interferometry, although the form factor to meet manufacturing requirements will likely be different. New metrology tools may incorporate multiple technologies to accomplish the needed measurements. But during this process each

metrology tool will generate its own systematic errors, so harmonizing multiple measurement techniques must be applied when using different methods to measure an identical sample. Thus, it is important to establish measurement standards to calibrate the results obtained by using different methods.

New classes of tools will be designed specifically for mass production supporting rapid set-up (calibration), reconfiguration for other uses, and easier use by manufacturing personnel. Manufacturing data will be received in real-time allowing for fast analysis and transformation into information and knowledge for downstream applications. In addition, tools will be available that can be configured in mass arrays, support extremely fast measurements, occupy limited production floor space, allow suitable manufacturing work volume, and be purchased at reasonable costs. Also, tools and process operations that require stringent environmental control and isolation (vacuum, vibration, temperature, particulate, etc.) will have new supporting equipment such as minichambers to lower the start-up costs for manufacturing at the nanoscale. As quality control during scale-up production becomes an issue, fault-tolerant design may be taken into consideration to enhance the production yield.

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

The semiconductor industry is already performing volume manufacturing of chips with features well below 100 nm; the ITRS provides a snapshot of currently available technology [1]. The ITRS indicates that some metrology tools have a “resolution” that is under 1.0 nm today and calls for this resolution to fall below 0.06 nm by the year 2018. It should be noted that the terms “resolution,” “accuracy,” and “precision” do not always have a common meaning within the industry. For example, the “resolution” of a Critical Dimension Scanning Electron Microscope (CD SEM) tool (currently used in semiconductor fabrication) generally means ability to differentiate between a line of a certain width and a line that is slightly wider. In contrast, the “resolution” of a research scanning electron microscope is defined as the smallest feature it can resolve.

The ITRS reports that tools are adequate for manufacturing today, but that 5–10 years down the road “no known solutions” are available for many critical metrology tasks. Revolutionary and not just evolutionary instrumentation may need to be developed. The semiconductor industry is primarily interested in tools capable of measuring high-value electronic parts in high-volume factories. Such tools may be of limited usefulness in other nanotechnology industries.

For imaging, industry currently uses tools such as optical microscopes, SEMs, transmission electron microscopes (TEMs) and scanning probe microscopes (SPMs). Linewidth measurements are performed by CD SEMs, CD atomic force microscopes and optical scatterometry tools. Special tools also exist for measuring pattern distortion and overlay.

The broad range of tools and applications makes a complete assessment of the state of the art for nanomanufacturing difficult. A summary of some available technologies is shown in Table 6.1. Recent advances in aberration-corrected TEMs have resulted in resolutions below 0.1 nm, although these tools are applicable to research and a limited set of manufacturing applications. For SEMs and CD SEM in manufacturing the primary problem is not resolution but image artifacts such as charging or scattering of secondary electrons. Improved modeling, better standards, and new technology such as variable pressure environmental SEMs may resolve some of these issues. Although scanning tunneling microscopes have achieved resolution below 0.1 nm, they are not as effective for samples with any topography. Atomic force microscopes and CD atomic force microscopes are limited by the size and variability of tip geometry, which can cause large artifacts.

In addition to improvements in these tools, completely new tools based on field ion emission or electron and atom holography may be necessary.

Table 6.1
Instrumentation for Nanomanufacturing

Scanning Probe Based	Beam Based	Photon Based	Enabling Technologies
Measurement approaches involving physical probes that either contact the sample directly or are controlled to be in near contact with the surface • Magnetic force microscope • Magnetic resonance force microscope • Chemical AFM • Dynamic force microscope • Cantilever sensors	*Measurement approaches using particle beams interacting with the sample for determination of the property or properties of interest* • Scanning transmission electron microscope (STEM) • Electron energy-loss spectrometer (EELS) • Scanning electron microscope (SEM) • Aberration-corrected transmission electron microscope (TEM) • Focused ion bean (FIB)	*Measurement approaches using photons of various energies ranging from optical to X- and gamma ray sources* • Scanning near-field optical microscope • Scanning interferometric apertureless microscope • X-ray or gamma-ray scattering or diffraction methods • Photonic force microscope • Scatterometry	• Computer software - Control and feedback for motion, alignment, and attachment control - Image correction - Data acquisition - Statistical process control - Autoalignment and registration • Nanometer scale positioning, manipulation and scanning • Actuators

GOALS, BARRIERS, AND SOLUTIONS

Many emerging 3D nanomanufacturing systems integrate several layers of functionality including sensing, actuation, control, and miniaturized devices (MEMS, NEMS, Bio-MEMS, micro and nanofluidic channels) for controlled deposition and removal of tiny amounts of material on various substrates. The applications for these nanomanufacturing systems range from multifunctional engineering systems to combinatorial chemistry to proteomic analysis. The enormous complexity of these systems demands that each layer is designed efficiently and optimally and that the various layers be integrated seamlessly.

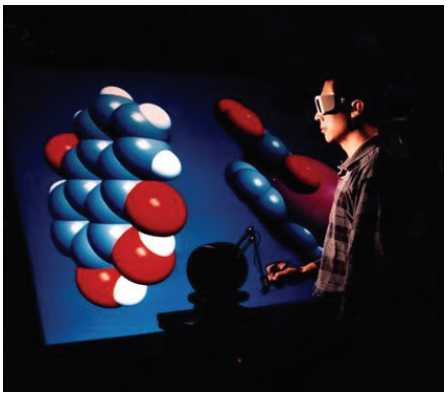

Figure 6.1. Virtual environments can provide nanomanufacturers with a way to link theoretical models with experimental measurements, enabling real-time decision support (courtesy of NIST Manufacturing Engineering Lab).

Direct prototyping of nanomanufacturing systems, involving several design and manufacturing cycles, can be error-prone and expensive. Modeling and simulation tools to enhance and drive the interpretation of measurements for nanomanufacturing can play an important role in enabling rapid computational prototyping and providing real-time decision support for nanomanufacturing systems. Developing modeling and simulation tools for nanomanufacturing systems is not trivial and involves several challenging aspects that range from fundamental definition of material properties to basic physical theory and behavior.

To achieve commercial success, companies need to have the ability to identify and measure key attributes of nanotechnology-based products such as those listed in Table 6.1. Thus, cross-cutting problems must be formulated and innovative solutions proposed. To establish a starting point and

help set the course for future work, three metrology and instrumentation challenges for nanomanufacturing are as follows:

1. Development of new instrumentation that has the ability to measure three-dimensional structures over large areas such as wafers used in the semiconductor industry
2. Development of new instrumentation that has the ability to characterize dense quantities of nanoelements under manufacturing conditions and in manufacturing-relevant time spans
3. Development of new software applications and sensor sets that have the ability to acquire and analyze large amounts of manufacturing data and make timely decisions to maintain manufacturing process control

Important research areas include the following:

- *Material properties at nanoscales:* computational models require material properties that are difficult to obtain at the nanoscale. The geometry factor may dramatically influence nanoscale material properties, and theoretical studies will provide insight into these effects. New approaches to characterize material properties at the nanoscale could result. Judiciously combining modeling and simulation with well-defined experiments to determine nanoscale material properties is critical.
- *Modeling and simulation at the nanoscale:* modeling and simulation of nanomanufacturing components requires the use of physical theories that can accurately predict device or component behavior. At nanoscale dimensions, physical phenomena governed by quantum mechanical motion of atoms and molecules become important and classical macroscopic

Table 6.2
Key Challenges and Barriers for Nanomanufacturing

Metrology	Challenges/Barriers
Real-Time Decision Support for Manufacturing	• Properties and theories for the macroscale must be redefined for the nanoscale • Multiscale computational methods that combine dynamics (molecular, coarse-grained mesoscopic and stochastic) and continuum theories • Complexity of integrating and analyzing large arrays of devices/components • Computational capabilities (high-speed and parallel processing, at source calculations)
"Full Device" Inspection With NM Resolution	• Defining a single set of requirements for substrate stages • Inspection tools with an overall precision and accuracy of ≈0.1 nanometer (corresponds to stage error on order of 10 pm) • Susceptibility of stage accuracy to environmental disturbances (atmospheric temperature, humidity and pressure fluctuation, vibration, acoustic, and electromagnetic disturbances)
Metrology for Liquid-Phase Nanomanufacturing	• System measurements vs. nanoelement (interfacial effects, influence on contacts on measurement) • Scalability of manufacturing processes to commercial production rates (loss of quality, environmentally unfriendly processes) • Very polydisperse, complex systems • Real-time monitoring of size and surface structure of growing quantum dots in colloidal dispersion • Visualization of nanoparticles in biological systems with resolution below and above the 100 to 200 nm scales in the liquid phase with non-destructive methods

theories can produce inaccurate results. As a result, the identification and development of accurate physical theories at nanometer scales is critical for providing real-time decision support as well as designing new 3D nanomanufacturing systems.

- *Multiscale modeling:* almost all nanoscale manufacturing components and processes exhibit some multiscale behavior. One of the greatest challenges in computational nanotechnology is the development of multiscale methods combining molecular dynamics, coarse-grained mesoscopic and stochastic dynamics and continuum theories for modeling of nanometer scale material, mechanical, fluidic and other components encountered in nanomanufacturing systems.
- *Reduced-order models:* one of the key features of nanomanufacturing systems is the integration of large arrays of mechanical, electrical, chemical, biological, and optical devices (referred to as mixed-technology manufacturing components) to perform a wide variety of functions. Continuum or multiscale analysis of large arrays of mixed-technology manufacturing components is crucial but can be expensive. Reduced-order or low-order models can play an important role in rapid analysis of large arrays providing real-time decision support.

Desirable Nanomanufacturing Instrumentation Attributes

Product throughput: Ability to make appropriate number and types of measurements to certify conformance of product to specifications.

Safety: Ability to make measurements without compromising the safety of the operator during final and work-in-process operations.

Footprint: Amount of production floor and work space that the instrumentation and supporting systems take up.

Tool accessibility to product: Ability of instrument to position the sensing probe precisely to make critical production measurements.

Sample preparation: Support minimal time differences between the time a product is produced to the time a sample is prepared for determining conformance.

Stage loading and presentation: Capability to quickly load and position stage for measurement of sample.

Vibration isolation: To conduct production measurements without vibration isolation.

Vacuum: To conduct production measurements without vacuum.

Thermal isolation or temperature control: To conduct production measurements without thermal isolation.

Setup: Minimize the number of set-ups and the time allotted to make measurements.

Maintenance: In production mode, the instrumentation should not result in more frequent servicing than the product production equipment.

Working volume: Larger working volumes translate to fewer set-up and registration operations and higher confidence in measurements.

Operator ease-of-use: Instrumentation can be set-up, calibrated, and run by trained personnel. Instrumentation should provide alarm for servicing (contaminated probes, etc.).

Flexibility: Instrumentation can be adapted or reconfigured to other uses with minimal effort.

Process Flow: Metrology operation does not interrupt product flow and preferably no off-line measurements are required.

- *System-level description:* system-level models and descriptions integrating nanomanufacturing components, sensors, actuators, and control units can provide invaluable information for real-time control and decision support.
- *Data analysis and visualization:* real-time data analysis and visualization of data are important tools to access the performance and accuracy of nanomanufacturing systems.

Nanomanufacturing will require rapid *positioning and measurement of planar patterns and 3D structures* for process development and control. "Planar patterns" include structures formed by planar fabrication processes such as lithography or self-assembly or formed by dispersed nanowires or other nanoparticles that have been fixed to a flat surface for the purpose of inspection. Rapid imaging and characterization of 3D structures and topography on the surface, which may be in the nanometer to micrometer scale in the vertical direction, will also be essential.

Typical measurements include device imaging, defect inspection and characterization, feature geometry and roughness, pattern distortion and overlay, atomic and chemical composition, magnetic and optical properties, and surface functionalization. New tools are needed to perform these required inspections at the subnanometer scale. Innovative probe design to measure physical, chemical, and mechanical properties is critical for all metrology applications, although manufacturing environments will present a unique set of constraints. Probes will likely be subjected to more extreme conditions (thermal, vibration, particulates), and time-dependant factors such as wear and fouling will have to be addressed. In some cases, the accuracy of the probe may be less important than its ability to rapidly recognize some feature of the product. An example might be a high-speed screening method fine-tuned for evaluating custom-designed nanostructured material.

Also, classes of probes will have to be capable of measuring component features as they move along on continuous production such as reel-to-reel material feed systems. In conjunction with the probe development, new compensation algorithms must be defined that provide the user with an accurate image of the scanned region, enabling knowledgeable engineering decision support.

A critical component of any inspection system is the substrate stage. Both fabrication (e.g., positioning, patterning) and inspection will require accurate substrate stages that can handle large substrates (e.g., up to 450 mm diameter for electronics). For example, although stages for STMs are capable of 0.1 nm resolution, they have severe limitations on substrate size and stage velocity. Because of the broad range of inspection and lithography applications contemplated, it is difficult to define a single set of requirements for substrate stages for nanomanufacturing.

Figure 6.2. Single-walled nanotubes with ~1.4 nm diameter. Developing effective metrology that enables in-process measurements allows companies to take an important step toward achieving predictable product properties (courtesy of Paul McEuen, Cornell University).

Generally speaking, stages will be required that assist in rapid patterning and inspection of sub-10 nm lithographic features and ≈1 nm single-wall carbon nanotube devices over large areas (on the order of 450 mm). Because the smallest features are on the order of 1 nm, the inspection tool needs to have a precision and accuracy of ≈10 % of this feature size, or ≈0.1 nm. Because this error must be apportioned between the imaging system, the stage, and unavoidable measurement artifacts, the stage error should be on the order of 10 pm.

Another critical need is metrology for *liquid-phase manufacturing of nano-based materials.* Examples of nanostructures assembled in nonaqueous liquid media include hollow nanospheres, colloidal nanoparticles (light-scattering quantum dots, gold nanoparticles), photonic crystals, SWNTs, molecules, DNA, and proteins. Although final structures are normally in the solid state or suspension phase, the functionality of these nanostructures is controlled by liquid-phase processing, and accurate metrology for this stage is critical.

Metrology for nanotechnology-based materials manufacturing should be developed for each of three stages of the manufacturing process: (1) quality control of the initial reagents, (2) metrology of the physical/chemical processes occurring during the liquid phase, and (3) metrology to ensure the solid material meets manufacturing specifications.

The first and third stages are linked strongly with nanocharacterization (Chapter 2). However, the reagents and nanoelements will be transported in the liquid phase, and consequently liquid phase and online monitoring will be essential. Manufacturing metrology for the liquid phase is important for the production of nanoelements as well as processes in which nanoelements are used to produce nanobased composites. Examples of nanoelements are quantum dots, carbon nanotubes, and nanowires made of conventional semiconducting materials, clays, or other materials.

Liquid-phase production and assembly present unique challenges to current manufacturing metrology tools. Online size monitoring and control to within some standard (~5% to 10% variation) will be required. Either direct or surrogate metrology to monitor and control chemical and physical properties (e.g., electrical, thermal, mechanical, chemical, defect structure) of nanoelements will also be essential. For example, in carbon nanotubes, important properties are length, diameter, chirality, defect structures, purity (absence of catalytic material and amorphous carbon), and mix of single-wall and multiwall species. For nanocomposites, a subset of metrology tools will be required for quality control of nanoelements as they enter and progress through the nanotechnology-based composite manufacturing process.

Laboratory-scale techniques are now available for making small quantities of nanoelements or nanostructures with the required perfection, but such techniques are not easily scalable to the production rates necessary for commercialization (see Fig. 6.2). Frequent problems encountered in scale-up include loss of quality (e.g., excessive polydispersity, low yield, excessive disorder) and processes with potential environmental impacts. Examples include quantum dots produced in trioctyl phosphine oxide solvent and other starting materials, such as methyl selenide, that are toxic and expensive.

A key challenge is to develop new methods and adapt classical ones for monitoring the structure of nanoscopic elements and ordered, partially ordered, or disordered assemblies of such nanoelements in the liquid state, possibly under process conditions (e.g., presence of flow, thermal gradients, electric and magnetic fields). Specific challenges include the following:

Nanomanufacturing Technologies and Methodologies

Contact Assembly

- Nano-tweezers
- Touch-probe

Non-contact Assembly

- Optical tweezers
- Magnetic tweezers

Directed Assembly

- Ink jet or droplet
- Continuous flow assembly
- External imposed force (e.g., magnetic, laser, electric)
- DNA-directed assembly

Self-assembly

- Functionalized surface
- Reactive surface
- Surface feature

Nanolithography

- Optical/Electron/Ion
- Dip-pen
- Scanned probe oxide (SPO)
- Nano-template with enzyme inks
- Nano-imprint

- *Scattering in SWNTs:* where systems are highly polydisperse in length and diameter, they may include other components with interfering length scales (e.g., surfactant micelles and polymers) and the length scales of assemblies can be comparable to the length scales of the nanostructures (e.g., size scales of mesophases in SWNTs in acids).
- *Real-time monitoring of the size and surface structure:* monitoring of the growing quantum dots in colloidal dispersions for online control of process parameters (e.g., reactor temperature, monomer concentration, anhydrous oxygen-free inert gas environment, organometallics of precursor compounds in nonaqueous media, stepwise reactions of coating core shells with metal sulfide or other coating compounds, surface derivations for biocompatibility), which will permit the growth of monodisperse samples and enable application in nanobiotechnology.
- *Visualization of nanoparticles in biological systems:* visualization with resolution below and above 100 to 200 nm in the liquid phase with nondestructive methods will require low radiation intensity, new detectors, and the ability to traverse thick samples.
- *Effect of flow on the orientation, aggregation/dispersion, and phase behavior:* the effect of flow on SWNTs and other nanoadditives in surfactant-stabilized suspensions, acid solutions, and polymeric dispersions.

The fluid-to-solid phase processing of nanomaterials for the manufacturing of functional mesoscale or larger composites may involve numerous constitutive steps, such as dispersion of nanomaterials in fluid phase matrices and self-assembly or directed self-assembly of nanomaterials into larger structures in the fluid or soft matrix phases. Computer modeling and simulation of the fluid-to-solid phase can play a significant role in guiding, controlling, and accelerating processing, as well as characterizing the final composite.

Theoretical modeling and simulation play a significant role in process and product design and prototyping before or during the experimental stage to shorten the R&D cycle. Modeling and simulation of dispersion and self-assembly phases can also lead to the discovery of entirely new behavior and potentially uncover new neat (pure or undiluted) or composite materials or applications. Key goals involve static and dynamic description of internanomaterial and nanomaterial–matrix interfaces at multiple lengths and timescales during the processing steps, as well as characterization of physical or chemical properties in processing steps and the final product.

The metrology of dispersion and self-assembly stages in the fluid phase may involve experimental measurement and computer simulation of linear or nonlinear response of the composite materials to the external optical, acoustic, electromagnetic, or thermal fields. Metrology of the fabricated nanocomposite material in the solid phase may involve physical surface mapping, indentation, and fracture, in addition to response behavior to external thermal, electromagnetic, and strain fields.

Nanoscale Elements

- Quantum dots
- Nanoparticles (powder and colloids)
- Nanocrystals
- Nanowires, nanofibers
- Nanoropes made up of dense quantities of nanoelements
- Nanofilms and other nanocoatings
- Nanopowder dispersions and suspensions
- Nanocomposites and fabrics
- Nanostructures (2D and 3D)
- Nanoporous structures
- Biomolecules such as proteins

Packaging of Nanoelements

- Aerosols

R&D INVESTMENT AND PRIORITY RESEARCH NEEDS

Three priority challenge topics have been developed to overcome the technical barriers and achieve the vision and goals for nanomanufacturing instrumentation and metrology. These challenge topics are summarized below and described in more detail in Priority Topics 6.1–6.3. They are not ranked in order of priority.

1. *Real-time decision support for nanomanufacturing (models, theory and experiment):* emerging 3D nanomanufacturing systems integrate several layers of functionality for deposition and control of small amounts of material on various substrates. The complexity of these systems requires considerable time and investment for prototype development. Modeling and simulation tools are needed to enable rapid computational prototyping and provide real-time decision support for manufacturing systems.
2. *"Full device" inspection with nanometer resolution (surfaces and 3D):* research is needed to develop tools for rapid positioning and measurement of planar patterns and 3D structures for the purpose of process development and control. Key technologies will include rapid imaging and characterization of 3D structure and topography on the surface. Stages will be required that assist in rapid patterning and inspection of sub-10 nm lithographic features, and ≈1 nm single-wall carbon nanotube devices.
3. *Metrology for liquid-phase manufacturing of nanomaterials:* metrology tools are needed for in-process control and monitoring of liquid-phase physical and chemical properties for nanoelements and nanocomposites. Quality control, reproducibility, scalability, and real-time monitoring are of primary importance. Modeling and simulation will have a key role in understanding all phases of processing and are important for fluid-to-solid phase processing of nanomaterials.

Figure 6.3. The 300 mm copper low-k dielectric process bay at the Advanced Tool Development Facility (ATDF), SEMATECH in Austin, Texas (courtesy of SEMATECH).

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

Historically, support for metrology research at the nanoscale has come from specific industries or consortiums such as the Semiconductor Research Corporation or SEMATECH, which are aimed at solving the short-term problems of the semiconductor industry. For this reason there has been little university educational and research infrastructure dedicated to long-term instrumentation and metrology research.

Universities should be encouraged to build up educational programs, and new Federal funds should be provided to allow university labs to upgrade facilities to enable nanometrology research. Three-way collaborations between university labs, Federal labs, and industry partners should be encouraged. Ample forums exist for the exchange of research, such as the annual meetings of the American Society of Precision Engineering and the International Conference on Electron, Ion, and Photon Beam Technology and Nanofabrication. However, the community would also benefit from an annual symposium involving all parties receiving NNI nanometrology research funds. This would enable researchers and program managers to meet in an open forum and would improve the quality of research management and review.

IMPLEMENTATION STRATEGIES

The manufacturing of nanobased composites is an industry in its nascent stage of development. For example, production of carbon nanotubes is measured in grams/month, and the size of composite materials is measured at most in centimeters. Large-scale production of nanobased composite materials will need to be measured in metric tons and miles of fibers. This scale-up is critical to industrial use of structural materials in new applications such as the aerospace industry, which would like to use carbon nanotube fibers for lightweight, high-strength composites. To achieve future manufacturing volumes, it is suggested that institutions currently producing lab-scale lots should be encouraged to start implementation of research and development on manufacturing processes for scale-up. This concept should be encouraged across the membership of the NNI. As an incentive, Federal funding should leverage existing funding focused on developing specific mission-driven nanotechnologies to hasten the implementation of manufacturing metrology that would be created by those leading laboratories where nanobased composite manufacturing processes are being developed.

Key research strategies include the following:

- Conduct a targeted R&D effort that is open to university, industrial and government laboratories with specific dollar amounts per year designated for nanomanufacturing activities. Collaborations between the three entities should be encouraged, but not required.
- Extend capabilities of key nanomanufacturing centers (e.g., instrumentation and metrology) to a broader set of researchers through shared facility services.
- Institute university programs in manufacturing that highlight the need for scale-up of nanoengineered products.
- Develop defined methods, procedures, and standards to interconnect nanoelements with micrometer-size elements. This would include the material properties for the connecting elements.
- Support the ability to view and measure manufacturing operations while they occur (real-time) and use this information for human-in-the-loop or computer control.

Priority Topic 6.1. Nanomanufacturing Grand Challenge
Real-Time Decision Support for Nanomanufacturing (Theory, Models and Experiment)

VISION AND GOALS Many emerging 3D nanomanufacturing systems integrate several layers of functionality for deposition and control of tiny amounts of material on various substrates. The enormous complexity of these systems and the large time and financial investment of developing prototypes creates a need for modeling and simulation tools to enable rapid design and prototyping and real-time decision support for nanomanufacturing systems.

KEY PERFORMANCE REQUIREMENTS

- Rapid computation to enable real-time decisions in developing nanomanufacturing system prototypes
- Rapid deployment of accurate probes and associated compensation models

COMMERCIAL APPLICATIONS

Real-time decision support for nanomanufacturing will be used in:

- Reel-to-reel material feed systems
- Carbon nanotube production
- Multi-probe systems

CHALLENGES & BARRIERS

- Properties and theories for the macroscale are not applicable to the nanoscale and must be redefined for the nanoscale
- Development of multiscale computational methods that combine dynamics (molecular, coarse-grained mesoscopic and stochastic), and continuum theories
- Complexity of integrating and analyzing large arrays of devices/components
- Computational capabilities
  - High-speed and parallel processing
  - At source calculations

SCOPE OF R&D

- Combine modeling and simulation with well-defined experiments to determine material properties at the nanoscale
- Develop accurate physical theories at the nanometer scale
- Develop reduced-order or low-order models
- Develop system-level models that integrate nanomanufacturing components, sensors, actuators, and control units
- Real-time data analysis and visualization of data

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – collaborative research with industry and federal laboratories

RISK

LOW — HIGH

Technical Risk

High risk, many uncertainties

Commercial Risk

High risk, many uncertainties

IMPACTS

LOW — HIGH

Opens venues for new device designs

Could lead to quantum computation

IMPLEMENTATION STRATEGIES A key strategy to addressing the need for real-time decision support for nanomanufacturing will be cost-shared R&D funding.

DEVELOPMENT TIMELINE

2005	2010	2015
Initiate R&D partnerships; prioritize and formulate problems to address	Effective simulation tools developed for nm instrumentation; instrumentation developed	Commercially available

Priority Topic 6.2. Nanomanufacturing Grand Challenge "Full Device" Inspection with Nanometer Resolution (Surfaces and 3D)

VISION AND GOALS The ability to rapidly position, measure, and characterize planar and 3D structures on the nano-to micron-scale is essential for process development and control in nanomanufacturing.

KEY PERFORMANCE REQUIREMENTS

- Rapid positioning and measurement of planar patterns and 3D structures
- Off-line and in-line measurement tools
- Rapid imaging and characterization of 3D structures and surface topography in the nanometer- to micron-scale in the vertical direction
- Accurate substrate stages that can handle large substrates (up to 450 mm diameter)

COMMERCIAL APPLICATIONS

The ability to measure 2D and 3D nm structures will find use in several nanomanufacturing applications, including:

- Defect inspection and critical dimension measurement
- Semiconductor chip design and fabrication
- New storage and computing device measurements
- Embedded structures measurement

CHALLENGES & BARRIERS

- Difficulty in defining a single set of requirements for substrate stages for nanomanufacturing
- Developing inspection tools with an overall precision and accuracy of approximately 0.1 nanometer, which corresponds to stage error on the order of 10 pm
- Susceptibility of stage accuracy to environmental disturbances (atmospheric temperature, humidity and pressure fluctuation, vibration, acoustic, and electromagnetic disturbances)
- Low cost for entry into nanoscale product market

SCOPE OF R&D

- Develop new materials with maximized stiffness and a minimized coefficient of thermal expansion
- Improved air and mag-lev bearings and low-noise motors
- Enhanced methods for heat removal at the substrate stage level
- Interferometer or optical encoder technology with higher resolution over long travel lengths and high velocities
- Improved methods to control environmental disturbances

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D

Industry – cost-shared R&D

Federal Laboratories – collaborative research with industry and universities

Universities – collaborative research with industry and federal laboratories

RISK

LOW — HIGH

Technical Risk

High risk, many uncertainties

Commercial Risk

High risk, many uncertainties

IMPACTS

LOW — HIGH

Opens venues for new device designs

Could lead to quantum computation

IMPLEMENTATION STRATEGIES A targeted nanomanufacturing R&D effort that is open to university, industrial, and government laboratories is recommended to drive technological advancements. Collaborations among the three entities should be encouraged, but not required and both NIST and NSF should be involved in managing the request for proposals and ensuing activities. In addition, funds should be available to universities to upgrade their facilities and support instrumentation and metrology research.

DEVELOPMENT TIMELINE

2005	2010	2015
Initiate R&D on materials and hardware, methods for heat removal	Development of interferometer or encoder technology	Commercially available instrumentation

Priority Topic 6.3. Nanomanufacturing Grand Challenge
Metrology for Liquid-Phase Manufacturing of Nanomaterials

VISION AND GOALS Liquid-phase manufacturing that is scaleable will play a key role in producing materials that are nanostructured by design. Nanoscale production presents unique challenges to current manufacturing metrology, and new tools are needed for monitoring and measuring physical and chemical properties and phenomena during the liquid phase.

KEY PERFORMANCE REQUIREMENTS

- Control/monitor size distribution of nanoparticles/quantum dots (5 % to 10 % variation), nanotubes (length, diameter, chirality, defects), and nanowires
- Property/functionality (electrical, thermal, mechanical) of nanoparticles and nanotubes
- Control/measure surface functionality, size distribution, jamming transition/flow of nanopowders/nanogranular materials
- Control dispersion and orientation of nanostructured materials (polymer nanocomposites, nanofibers, as extruded or molded); measurement of phase behavior and electrical, optical and thermal properties
- Soft disordered and interfacial systems: deconvoluted signals from polydisperse systems and complex environments
- Ability to characterize nanostructured materials under non-equilibrium or unsteady state conditions

COMMERCIAL APPLICATIONS

Manufacture of:

- Nanoelements (nanoparticles (NP) such as quantum dots, carbon nanotubes (NT), nanowires (NW))
- Nanostructured materials such as polymer nano-based composites and nanofibers
- Nanopowders and nanogranular materials for pharmaceutical and geological applications

CHALLENGES & BARRIERS

- System measurements versus nanoelement (interfacial effects, influence on contacts on measurement)
- Scalability of manufacturing processes to commercial production rates (loss of quality, environmentally unfriendly processes)
- Very polydisperse, complex systems
- Scattering in single-walled carbon nanotubes
- Real-time monitoring of the size and surface structure of the growing quantum dots in colloidal dispersion for online control of process parameters
- Visualization of nanoparticles in biological systems with resolution below and above the 100 to 200 nm scales in the liquid phase with non-destructive methods

SCOPE OF R&D

- New metrology tools for in-process control and monitoring of liquid-phase physical and chemical properties (size distribution, electrical, thermal, mechanical properties, purity (absence of catalytic materials and amorphous carbon), chirality, defect structure, surface functionality, dispersion, orientation, phase behavior)
- New metrology tools for quality control of nanoelements as they enter nano-based composite manufacturing
- Materials that report on their own state (self-reporting)
- Techniques for characterizing dense collections of nanoelements under manufacturing conditions (dimensions, static and dynamic properties)
- Modeling and simulation of dispersion and self-assembly phases
- Static and dynamic models of inter-nanomaterial and nanomaterial-matrix interfaces at multiple length and time scales during processing

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – cost-shared R&D
Industry – cost-shared R&D
Collaborative research efforts with Federal laboratories, industry and universities

RISK

	LOW	HIGH
Technical Risk		
Commercial Risk		

IMPACTS

	LOW	HIGH
Enabling materials manufacture		
Control of materials quality		

IMPLEMENTATION STRATEGIES Institutions currently producing lab-scale lots and manufacturing processes should be encouraged to implement R&D of manufacturing metrologies. Incentives could leverage efforts and accelerate development of metrologies at laboratories where nano-based composites and manufacturing technologies are under development.

DEVELOPMENT TIMELINE 2005 2010 2015

- Metrology tool development for properties control
- Supporting activities in modeling and simulation

SUMMARY

Reliable, reproducible nanomanufacturing supported by rapid, accurate metrology and instrumentation is the key to achieving the economic potential of nanotechnology. To achieve this goal, existing metrology tools need to be dramatically improved, and innovative tools based on entirely new ideas will need to be developed. Metrology will need to move out of the laboratory and onto the manufacturing floor, where it cost-effectively provides rapid analysis of all aspects of processing and is usable by manufacturing personnel. There are possible synergies with the semiconductor industry goals (ITRS) that could advance metrology and instrumentation, but these are limited to certain applications and will not solve all nanomanufacturing metrology challenges. A combination of efforts in government laboratories, industry, and universities will be needed to develop the essential tools needed for nanomanufacturing. Support for university education and research in nanomanufacturing is essential for future development of this emerging industry. Metrology tools will be needed for all phases of manufacturing, from synthesis to determination of final product quality.

Communication and collaboration among researchers involved in nanomanufacturing metrology and instrumentation research and development will be critical to accelerate progress and disseminate information on breakthroughs and advances. It is recommended that an annual symposium of NNI-supported nanometrology research be held for this purpose. This will provide a means for raising awareness of the larger manufacturing community that metrology and instrumentation are essential pieces for succeeding in manufacturing at the nanoscale.

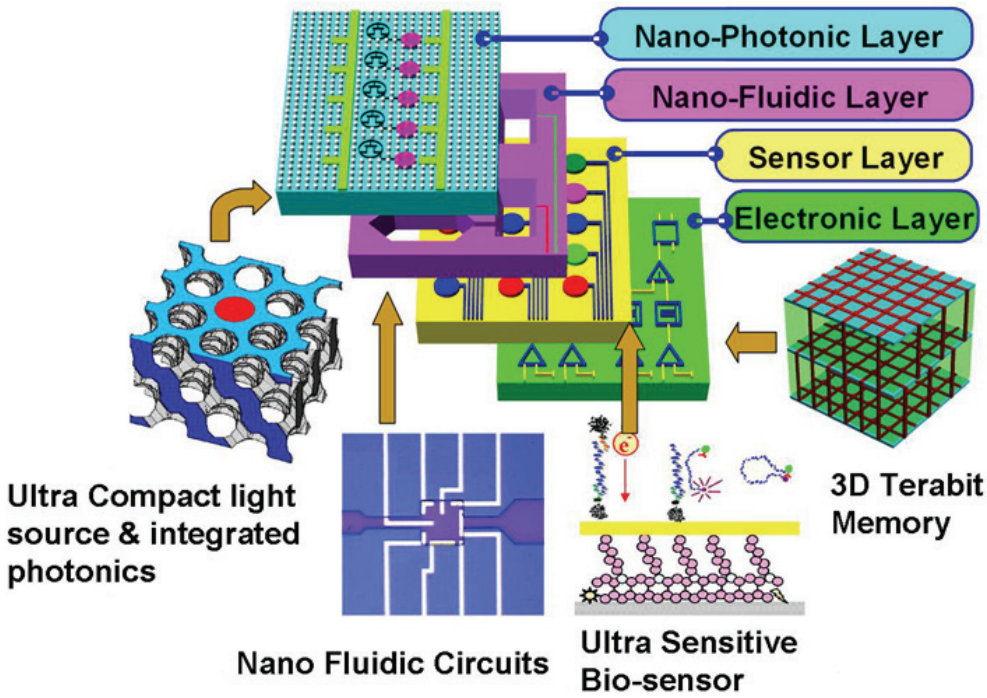

Figure 6.4. Potential future integrated nanoscale system that achieves functionality by taking advantage of phenomenon unique to specific size scales and domains (courtesy of Xiang Zhang, University of California).

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7. Computational Science Issues in Nanoscale Metrology

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SCOPE

Computation has emerged as a highly significant enabler for R&D in science and engineering, complementing and enhancing the traditional approaches of theory and experiment. Computational science refers to the development and application of informatics techniques and tools that support scientific discovery aided by computation. These include mathematical modeling, computer simulation, algorithms, data management, statistical analysis, and scientific visualization.

Computational science is expected to play a critical enabling role in nanoscale instrumentation and metrology, cutting across all areas considered in this report to enable greatly accelerated development of new measurement technologies. Here we identify key mathematical, statistical, and computational technologies and tools that will need advancement to support the development and exploitation of nanoscale instrumentation and metrology.

VISION

The techniques and tools of computational science will be critical enablers of all aspects of nanoscale metrology and instrumentation. The role of computational science now and in the future includes:

- Providing fundamental information and insight for nanometric tools and methodologies
- Aiding the interpretation of measurements at the nanoscale
- Providing virtual measurements of nanoscale features or properties
- Enabling fast, global access to real and virtual nanoscale measurements

Computational Science Vision for Nanotechnology

Reliable, validated models and simulations based on sound scientific theory, with the capability for

- *Generating fundamental information needed for nanometric tools and methods*
- *Generation of virtual nanoscale measurements*
- *Interpretation of nanoscale measurements*
- *Rapid global access to measurement data*

Reliable models based on sound, scientific theories will be needed to plan and interpret measurements, and simulations will be needed to test these theories, validate measurements, and even to stand in for measurements not yet possible. Indeed, in many instances computational predictions are currently more accurate, precise, and reliable than experimental measurements.

Already, the field of computational science is contributing to revolutionary advances in nanoscale science and engineering. To support nanoscale metrology and instrumentation, significant additional breakthroughs and improvements in many key areas of computational science, mathematics and statistics will be required over the next decade.

CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

Enormous advances in computing and communications hardware, mathematical methods, and software tools have occurred over the past 15 years. For example, computing speed has improved by nearly four orders of magnitude during this period, as evidenced by improvement in Gordon Bell Prize winners (1 Gflop/s in 1988 versus 5 Tflop/s in 2003). During the same period, advances in algorithms have been similarly impressive. Density functional theory, fast multipole methods, improvements in Monte Carlo techniques, adaptive multigrid techniques, and parallel linear algebra methods, have greatly extended the range of problems that can be successfully addressed by computation.

These advances have led to the emergence of computation as the third pillar of science, providing significant new capabilities for scientific discovery that complement the traditional approaches of theory and experiment. This new approach to science and engineering has led to remarkable productivity in the study of topics as diverse as global atmospheric dynamics and the human genome [1]. The success of National Science Foundation (NSF) supercomputer centers, and the widespread deployment of clustered computing systems supporting parallel scientific applications attests to the efficacy of this approach to scientific discovery.

Computational science has already had a significant effect on nanotechnology. For example, advances in theory and modeling that explained the microscopic quantum-mechanical processes responsible for the giant magnetoresistance effect were largely responsible for the unprecedented speed of its application to commercial hard disks and magnetic sensors in the late 1990s. More recently, NASA computer models predicted that carbon nanotubes could be made to function as transistors several years before such devices were created in the lab [2]. Such successes have led to recommendations for increased funding of computational nanotechnology [3].

Computational science also plays an important role in the development, operation, and analysis of modern measurement systems. Mathematical modeling and simulation are employed to provide fundamental understanding of the phenomena to be measured. In particular, they can characterize the measurement environment necessary for designing effective instrumentation. Theoretical models can become part of the instrument itself, providing a transformation from the quantity actually measured to the value desired. When measured results are imagery, mathematical deconvolution techniques can be applied to improve them. Finally, statistical uncertainty analysis is critical for understanding the validity of measured results.

Informatics is a critical component of most modern measurement systems. The tools of information technology are used to collect experimental data, transform it, transport it, and archive it. As a result, laboratory automation systems, signal processing tools, communication networks, and database systems are all found in modern measurement laboratories. Once data have been amassed, the tools of computational science are used to interpret them. Here techniques of statistical analysis, image processing, data mining, and visualization are invaluable.

In some cases physical measurement is simply impractical or impossible. In these cases virtual measurement (i.e., measurement based on modeling and simulation alone) may be the only recourse. Modeling and simulation allows study of phenomena impossible to create in a lab, or too difficult, expensive, or dangerous for experimentation (like nuclear weapons testing). A goal of the Department of Energy Accelerated Strategic Computing Initiative (ASCI) program is to develop the capability of doing the latter. Such work remains enormously challenging. If modeling and simulation are to be used in place of physical measurement, then rigorous verification and validation (V&V) of models and simulations must be done. However, formal V&V is rarely performed in research labs, and techniques for statistically sound uncertainty estimates for virtual

measurements are just beginning to emerge. Some professional societies have taken an interest; the American Institute of Aeronautics and Astronautics (AIAA) has published V&V guidelines for computational fluid dynamics simulations [4], and the American Society of Mechanical Engineers is developing its own set of guidelines.

GOALS, BARRIERS, AND SOLUTIONS

The critical needs of nanoscale metrology and instrumentation for computational science lie in two general areas: (1) modeling and simulation coordinated with high-precision nanoscale measurements, and (2) management and exploitation of data from nanoscale measurement and simulation.

Close interaction between theorists and experimentalists will be necessary to develop an understanding of the nanoscale processes necessary to support measurement and instrumentation. Ideally, modeling and simulation efforts will be closely coordinated with high-precision measurement efforts, with models accurately representing the measurement scenario. Unfortunately, today, modelers and experimentalists are too often in separate camps, with modeling efforts disconnected from experimental efforts. This cultural divide hurts both groups, as simulation can play a crucial role in validating measurements, and measurements can play a crucial role in validating simulations. The development of interdisciplinary research teams centered on particular measurement domains and technologies would greatly accelerate progress in nanoscale metrology and instrumentation.

Significant progress in modeling and simulation technologies and tools will be necessary to address the difficult questions associated with nanoscale metrology. Modeling and simulation in this domain is far from routine, straining the existing state of the art in theory, mathematical methods, computational algorithms, and computing hardware.

A challenging aspect of nanoscale modeling and simulation is the enormous range of length and timescales that must be addressed (see Fig. 7.1). No modeling approach can adequately cover this range. For example, when considering scenarios with small numbers of atoms over a period of a

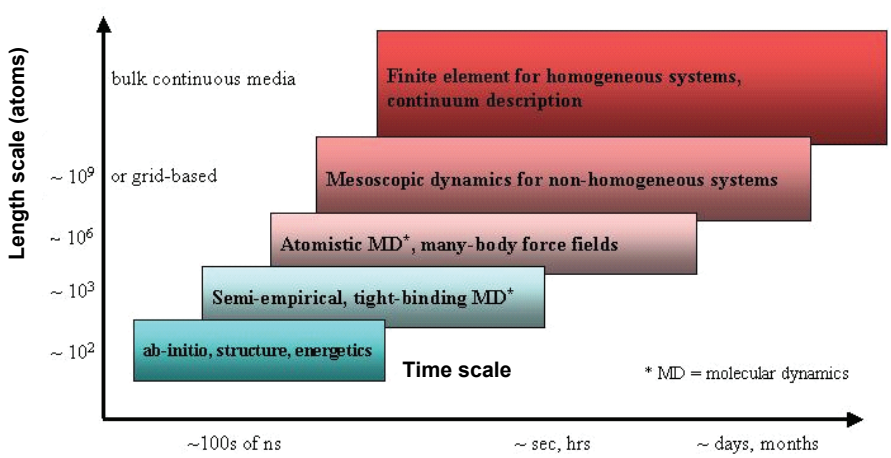

Figure 7.1. Nanoscale modeling spans a wide range of time and length scales, and a variety of modeling approaches are used, none of which can span all regimes. This figure provides a very rough indication of the applicability of various approaches (based on a figure provided by Deepak Srivastava of NASA Ames Center for Nanotechnology).

few nanoseconds, *ab initio* quantum mechanical electronic structure calculations can be used to capture the essential physics. However, such computations quickly become difficult when the number of atoms grows. For systems with 1,000 atoms, semiempirical tight-binding molecular dynamics simulations are more effective. For atoms numbering in the millions and timescales of seconds or minutes, atomistic molecular dynamics are used. In some cases the performance of a nanostructure must be studied over long periods of time, from days to months to years. The only computationally feasible approaches in this case are those that treat large numbers of atoms (e.g., 10^9) such as mesoscopic dynamics or continuum approaches. Each of these modeling and simulation regimes is, in itself, extremely challenging, both from the standpoint of developing reasonable models and solution methods, and from that of performing the resulting computations.

To treat all relevant time and length scales for a given problem related to nanoscale measurement, it often becomes necessary to use multiple modeling approaches. Here, techniques for transforming information from one type of simulation to another, such as homogenization for coarse-graining, are needed. Unfortunately, today these remain difficult to formulate, and methods used remain ad hoc. Hence, it is critical to develop techniques and tools to bridge time- and length scales for nanoscale modeling and simulation.

In each modeling regime there is a need to achieve as high fidelity as possible. This leads to a push for more atoms, more complete physics, 3D models, and time-dependent models. As a result, computational simulations can become very large, taxing the largest computers available. Further, if results are to influence experiments, they must be produced quickly. Unfortunately, today's algorithms and high-performance computers are inadequate. Optimally fast methods (i.e., methods whose execution time scales linearly with the size of the input) to enable *ab initio* calculations on larger numbers of atoms than currently possible are required to predict forces, structure, and optical, electronic, and mechanical properties. Acceleration algorithms are needed for advancing timescales of quantum and classical simulations of electron transport, self-assembly of nanoscale building blocks, and electronic, optical, photonic, mechanical and other properties of nanomaterials and nanodevices. Variants of these methods highly tuned to take advantage of modern parallel and distributed computer architectures are critically needed.

Another important aspect of modeling and simulation is verification and validation. Verification is the process of ensuring that computations correctly provide accurate solutions to the mathematical problems posed, and validation is the process of ensuring that models and simulations adequately represent the physical systems that they are modeling. Unfortunately, V&V remains difficult to perform because of the lack of benchmark problems and forums for systematic intercomparison of codes. Better understanding of error and convergence properties of numerical methods are needed, as well as the development of methods with computable *a priori* or *a posteriori* error measures. Methods for formal statistical uncertainty analysis of models and simulations must be developed. Close cooperation between experimentalists and theorists is critical for the success of such efforts.

Finally, theorists and experimentalists want tools for modeling and simulation that are easy to use. Well-engineered problem-solving environments should be developed to allow virtual experimentation, and ultimately virtual measurement, for nanoscale systems. As modeling and simulation techniques improve, it will become increasingly feasible to incorporate such models themselves into future instrumentation to provide real-time enhancement of physical measurement processes.

The second major area in which computational science plays a role in nanoscale measurement and instrumentation is in managing and exploiting measured results. The techniques and tools of information technology are already widely used in modern measurement systems. However, when dealing with length scales far below those at which we can naturally "see," one often finds the need

to collect significantly more data. Thus, nanoscale measurement often leads to petascale databases. The trend to larger datasets can also be seen in the development of high-throughput experimental techniques, and the prevalence of image data resulting from commonly used instruments such as scanning electron microscopes, atomic force microscopes, and so forth. Finally, when modeling and simulation are used to validate or even to stand in for physical measurement, one can easily obtain many gigabytes of data describing state variables at each point in space for each time step.

Dealing with massive datasets is challenging. Specialized tools must be integrated with instrumentation to collect, process, and off-load data for archiving. High-bandwidth data acquisition and transport are needed, supported by high-speed intralaboratory networks and distributed data processing techniques. Statistical process sampling for monitoring and control of instruments is needed. Techniques such as real-time statistical state estimation and stochastic signal detection, identification, and classification are critical. Interactive visualization tools are important in real-time control of instrumentation. By merging data collected in real-time both on the current state of the instrument as well as on the measured properties of the sample, one can construct an interactive virtual model that allows visualization of the measurement environment at the nanoscale and to control the measurement apparatus to perform a particular task. Immersive 3D visualization environments may be particularly effective here. Such tools are equally valuable to computational scientists who may want to interactively steer a simulation run. Such tools can also be used to remove geographic impediments to use of scientific instruments and computational models, as interfaces become Web-based for an increasingly distributed user base [5].

Mathematical and statistical tools are necessary for transforming data, reducing its size, correcting and improving it, and assessing its validity. Statistical design of experimental techniques can effectively reduce the amount of data needed to be collected, for example. At the single-molecule level, measurements contain essential noise, which cannot be abated with more refined experimental conditions. This makes system state identification and estimation fundamental to the concept of measurement meaning that measurement becomes computational in an essential way. Thus, statistical signal processing techniques are needed to transform data and separate signal from noise. Much data is collected in the form of imagery, and fuzzy, noisy imagery is common. New effective deconvolution techniques must be developed to improve collected data. Finally, the uncertainty and correlation in nanoscale measurement data must be carefully assessed, and the ever-increasing complexity of instrumentation systems provides challenges for existing statistical approaches.

Community data warehouses for long-term archiving and public access to nanoscale measurement data, similar to the successful Protein Data Bank, must be developed and sustained to provide infrastructure for continued scientific discovery. Standardized database schemas and metadata will be needed to enable effective search and retrieval of such massive nanoscale datasets, as well as its communication to other researchers. Especially necessary are tools for intelligent retrieval, data mining, and knowledge discovery in data tuned to the needs of nanotechnology researchers and developers. For example, methods of fusing or combining data from several instruments to provide understanding of a single sample are needed. Finally, systems for interactive (e.g., immersive) visualization of nanoscale data provide one of the best tools for scientific discovery in massive data sets, providing scientists with the ability to “see” what is happening; humans remain very effective at data mining if provided with the right tools.

R&D INVESTMENT AND PRIORITY RESEARCH AREAS

Although the effective exploitation of computational science for nanoscale metrology and instrumentation presents many challenges, they can be classified into two broad areas: (1)

modeling and simulation coordinated with high-precision nanoscale measurements, and (2) management and exploitation of data from nanoscale measurement and simulation (see Priority Topics 7.1 and 7.2).

SCIENTIFIC AND TECHNICAL INFRASTRUCTURE NEEDS

Computational science for nanoscale metrology and instrumentation relies heavily on advances in basic information technology infrastructure, especially in the area of high-end computing and communications technologies. Revolutionary improvements in high-performance computing and access to fast machines are critical for increasing the accuracy and precision of virtual measurements from *ab initio*, molecular and mesoscale simulation. Concerted improvements in algorithms, software, hardware (processor speed, memory access, etc.), and middleware will all be required in this regard. Significant improvements in high-speed communications will be required to support increased interprocessor communication and data transfer for distributed and parallel high-performance computing, computational steering, interactive visualization, data mining and manipulation, and database management.

IMPLEMENTATION STRATEGIES

The development of techniques and tools for computational science to support nanoscale metrology and implementation must be a cooperative effort of industry, government, national labs, and academia. Government should provide funding for precompetitive research, as well as for facilities such as supercomputer centers providing access to large-scale, high-end computer facilities. Academia should be a source for fundamental R&D, as well as for demonstrations of early concept tools. National labs should also provide fundamental R&D, as well as state-of-the-art facilities and resources. Examples of such resources are prototype tools, data centers, and forums and guidelines for verification and validation. Industry should provide guidance for R&D efforts, and ultimately should be responsible for the commercialization of techniques and software tools for nanoscale modeling and simulation.

In the near term, existing models will be expanded to increase capabilities and identify gaps. R&D to uncover and analyze new modeling methodologies and computational algorithms must continue. The best such methods must be embodied in software libraries, and eventually in prototype problem-solving environments for widespread use in the research community. Ultimately, the transition of such tools to the commercial sector is desired.

Mechanisms to encourage the formation of interdisciplinary research teams for nanoscale metrology that include experimentalists, theoretical scientists, mathematicians, statisticians, and computer scientists must be found. While addressing the needs of specific measurement projects, such teams will also serve to identify the critical informatics techniques and tools that will be needed to support nanoscale measurement more broadly. Training of a new species of interdisciplinary systems engineer that can deal with mathematics, statistics, parallel processing, data acquisition, databases, and computer systems must begin. Interdisciplinary teams will also be an important proving ground for emerging verification and validation methodologies. To further encourage the latter, open forums for verification and validation of models and simulations in various areas should be evolved. Ultimately, industry standards and best practices for verification and validation of virtual measurements must be developed.

Priority Topic 7.1. Nanoscale Computational Science Modeling & Simulation Coordinated with High Precision Measurements

VISION AND GOALS In the future, scientists and engineers will have access to highly accurate and reliable models and simulations, with the capability for generating fundamental information needed for the development of nanometric tools and methods. The supporting goal is to develop sound scientific theory and models, improved numerical methods and software, and a verification and validation infrastructure that enhances the impact of both theorist and experimentalist.

KEY PERFORMANCE REQUIREMENTS

- Close coordination between theorists and experimentalists
- Techniques and tools to attack problems across wide time and length scales
- High fidelity, reliable, efficient, and well-characterized computational results
- Well-engineered problem-solving environments (PSEs) to allow facile virtual experimentation and measurement

CHALLENGES & BARRIERS

- Inadequate existing state-of-the-art in theory, mathematical methods, computational algorithms, and computing hardware
- No single modeling approach to cover the enormous range of length and time scales
- Difficulty in using multiple modeling approaches in a single problem
- Very large size of computational simulations and required computing power
- Difficulty of verification and validation
- Disconnect between theorists and experimentalists

SCOPE OF R&D

- Models to relate atom-by-atom structure to device performance
- Models to interpret measurements
- Models and methods that support a broad range of time and length scales
- Fast algorithms for core computations
- Methods with *a priori* or *a posteori* error estimates
- Detailed inter-comparisons between measurements and simulations
- Uncertainty analysis for virtual measurement

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government - research funding, high end computing resources

Federal Laboratories - R&D, benchmark development, prototype tools

Universities - R&D, new methods

Industry - R&D partnerships, applications

Suppliers - commercialize tools

End Users - applications

RISK

LOW — HIGH

Technical Risk

Modeling problems very challenging

Commercial Risk

Prototypes tested in R&D labs

IMPACTS

LOW — HIGH

Use across many applications

Reliability of predictions

Relevance to commercial applications

IMPLEMENTATION STRATEGIES R&D in methods and tools performed in universities and Federal labs. Interdisciplinary teams of experimentalists and theorists are established. High-end computing resources provided by government. Joint development of verification and validation frameworks. Commercialization of tools.

DEVELOPMENT TIMELINE

2005	2010	2015
R&D in efficient mathematical methods, multiscale analysis	Well-established verification and validation frameworks in use	Commercialization of problem-solving tools

Priority Topic 7.2. Nanoscale Computational Science
Management & Exploitation of Nanoscale Measurement/Simulation Data.

VISION AND GOALS Advances in informatics will significantly enhance the acquisition and exploitation of nanoscale data, both from measurement and simulation. A supporting goal is the development of new techniques and tools for intelligent retrieval, data mining, and extracting knowledge from nanoscale data to enhance nanoscale metrology.

KEY PERFORMANCE REQUIREMENTS

- Distributed access and control of instrumentation
- High bandwidth data acquisition and transfer
- Large dataset management and analysis capabilities
- 3D immersive visualization of nanoscale data
- Improved image and signal analysis methods
- Characterization of uncertainty in nanoscale data
- Community warehouses for nanoscale data

CHALLENGES & BARRIERS

- Need to collect, process massive datasets from measurement and/or simulation
- Data difficult to interpret
- Image data collected is fuzzy, noisy
- Measurement uncertainties difficult to characterize
- Data from wide variety of instruments, for wide variety of properties
- Data difficult to exchange

SCOPE OF R&D

- Statistical design of experiments
- Statistical process sampling, signal identification
- Dynamic real-time processing of large arrays
- Deconvolution techniques to improve collected data
- Data fusing reconciliation, combining data from, e.g., ATM, SEM, STM
- Integrated tools for intelligent retrieval, data mining and knowledge discovery
- Interactive visualization of nanoscale data
- Uncertainty analysis
- Standardized database schemes and metadata

PARTNERSHIP ROLES AND RESPONSIBILITIES

Government – research funding, network access

Federal Laboratories – R&D, data centers

Universities – R&D

Industry – experimental validation

Suppliers – commercialize tools

End Users – applications

RISK

LOW — HIGH

Technical Risk

Methods must address wide application

Commercial Risk

Tools must be highly versatile

IMPACTS

LOW — HIGH

Current technology inadequate

Reliability of measurements

Use across many applications

Relevance to commercial applications

IMPLEMENTATION STRATEGIES R&D in methods and tools performed in universities and Federal labs. Computer scientists, mathematicians, statisticians team with instrumentation developers. Access to high-end computing networks provided by government. Tools commercialized by private sector. Data centers hosted by Federal labs.

DEVELOPMENT TIMELINE

2005		2010
R&D in data acquisition, analysis, visualization	Development of informatics-enhanced nanoscale measurement systems	Commercialization of data-mining tools

Formation of data centers for the archiving and distribution of nanoscale measurement data should be considered. Such centers could be the focal point for R&D in data-mining technologies, as well as for work on standardization of schemas and metadata to support data exchange.

SUMMARY

Computational science offers the potential for substantially improved understanding of processes necessary to support the development of measurement technologies and instrumentation systems at the nanoscale. In particular, significant acceleration of progress may be possible resulting from interdisciplinary collaboration and the close interaction of theory and experiment. In addition, the integration of emerging informatics techniques and tools will provide the cyber infrastructure necessary to develop highly capable nanoscale measurement platforms. However, attaining these goals will require substantial investments in research and development in theory, modeling, and algorithms, as well as the development of new informatics technologies and tools.

REFERENCES

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8. THE PATH FORWARD

Realizing the potential of emerging nanotechnologies will require a solid, supporting foundation in instrumentation and metrology. This report has outlined some of the most important challenges that must be addressed, as well as the research and development pathway to meet the metrology needs of a future commercial enterprise that depends on and benefits from nanotechnology-based materials and devices. Although much progress has been made, current instrumentation and metrology are nearing the limits of resolution, and new technology will be necessary to support an emerging and potentially enormous nanotechnology-based industry.

Developing effective funding strategies, supporting collaborative multidisciplinary research activities, demonstrating and validating technology, and nurturing the creation of a workforce skilled in nanotechnology are all critical elements in building the required new suite of instrumentation and metrology tools. However, it will be equally important to focus these efforts on the areas that are going to be the most critical to commercial producers of nanotechnology-based products, considering near-, mid- and long-term product and market potential.

The semiconductor industry has realized multiple benefits from the establishment of a well-funded and organized consortium. Great strides have been made in developing the supporting technology to meet the challenges and needs outlined in the ITRS. These advances have been made through the concentrated expenditures of millions of dollars and the evolution of generations of instrumentation. In the field of nanotechnology, however, it is early and such a structure or structures have not yet materialized. Manufacturing groups have yet to take steps to organize, and there is little focus among manufacturers about what the key products, applications, and common instrumentation and metrology needs will be.

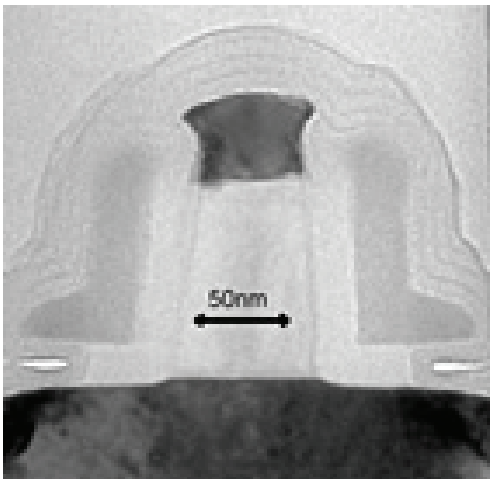

Figure 8.1. The transistors implemented in Intel's new chip making process, shown above, are among the smallest ever to be designed into a commercial microprocessor, measuring only 50 nanometers. Hundreds of these transistors fit inside a red blood cell (courtesy of Intel).

A crucial step in the path forward will be to gain consensus on what the focus should be for the near, mid, and long term. Commonalities among manufacturers should be identified so that research targets can be focused effectively. Specific avenues of promising research should be identified and grouped according to near-term, mid-term, and long-term goals. Applications that would be affected by successful research should be identified and prioritized in terms of future potential.

A generic technology roadmap, whether it is a national or international endeavor, much like the ITRS, is needed to provide such focus for the many diverse efforts in nanotechnology. Such a proposed "national technology roadmap for nanotechnology" (NTRN) or "international technology roadmap for nanotechnology" (ITRN) for instrumentation and metrology would define where the industry wants to be in 5, 10, and 15 years—and beyond. The roadmap should be dynamic, with experts coming together every 2 years or so to review progress and redefine goals and pathways.

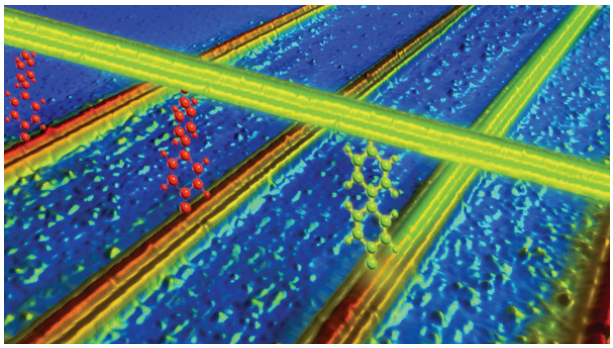

Figure 8.2. Artist's conception of a molecular electronic circuit. Molecular switches connect nanometer-scale wires in two different planes to form a cross-bar array (courtesy Madworks Concept Images).

A national roadmap would provide guidance for technology development as well as direction for instrument manufacturers in providing the metrology tools needed. Creating such a roadmap will require the identification of nanotechnology commonalities and subsequent focus on needs, as described above.

It is anticipated that the United States will be a world leader in the nanotechnology revolution, creating an array of new products in fields from electronics to medicine. The NTRN could give focus and direction to the future of this industry and more clearly define what is needed to ensure world leadership and a competitive edge. Instrumentation and metrology are a key component of these efforts—without them, manufacturers cannot go forward.

Strong, focused leadership will be required to make the national roadmap a reality. A consortium-type organization similar to SEMATECH, initially co-funded by government and industry, could serve as a focal point and champion for the national roadmap and could help to accelerate the needed development in instrumentation and metrology as well as other fields. To successfully develop a national roadmap, industry will need to come together to identify common goals, similar to the ITRS, and provide the needed input.

Summary of Recommendations

The overarching grand challenge derived from the workshop, which essentially summarizes all the individual challenges for the areas surveyed, is to develop the ability to image or measure any nanostructure for any relevant property in three dimensions with atomic accuracy. This requires the development of new metrology instrumentation and infrastructure for both laboratory research and nanomanufacturing. The priority research areas identified in this report are reiterated in Figure 8.3 for the important areas of measurement technology. These represent potential grand challenge topics for nanoscale instrumentation and metrology. In addition, broad-based recommendations to develop the instrumentation and metrology required to enable nanotechnology and the future manufacturing of nanotechnology-based products are:

- Develop a national (or international) technology roadmap for nanotechnology (NTRN) for instrumentation and metrology similar to the current International Technology Roadmap for Semiconductors to guide technology development and assist instrument manufacturers in providing the needed measurement tools within a reasonable lead time
- Develop strong educational programs and leverage Federal laboratories that address the development of measurement infrastructure and advanced measurement instrumentation; coordinate funding with agencies to provide effective support for program areas of joint interest
- Leverage national laboratories' user facilities to foster the development of new measurement techniques and development of a National User Facility for Nanometrology

- Foster the development of consortia cofunded by government and industry tasked to bridge the gap for the development of sector-specific instrumentation for nanometrology for nanomanufacturing
- Invest in integrated computational methods to develop predictive and assessment tools for nanometrology and nanomanufacturing

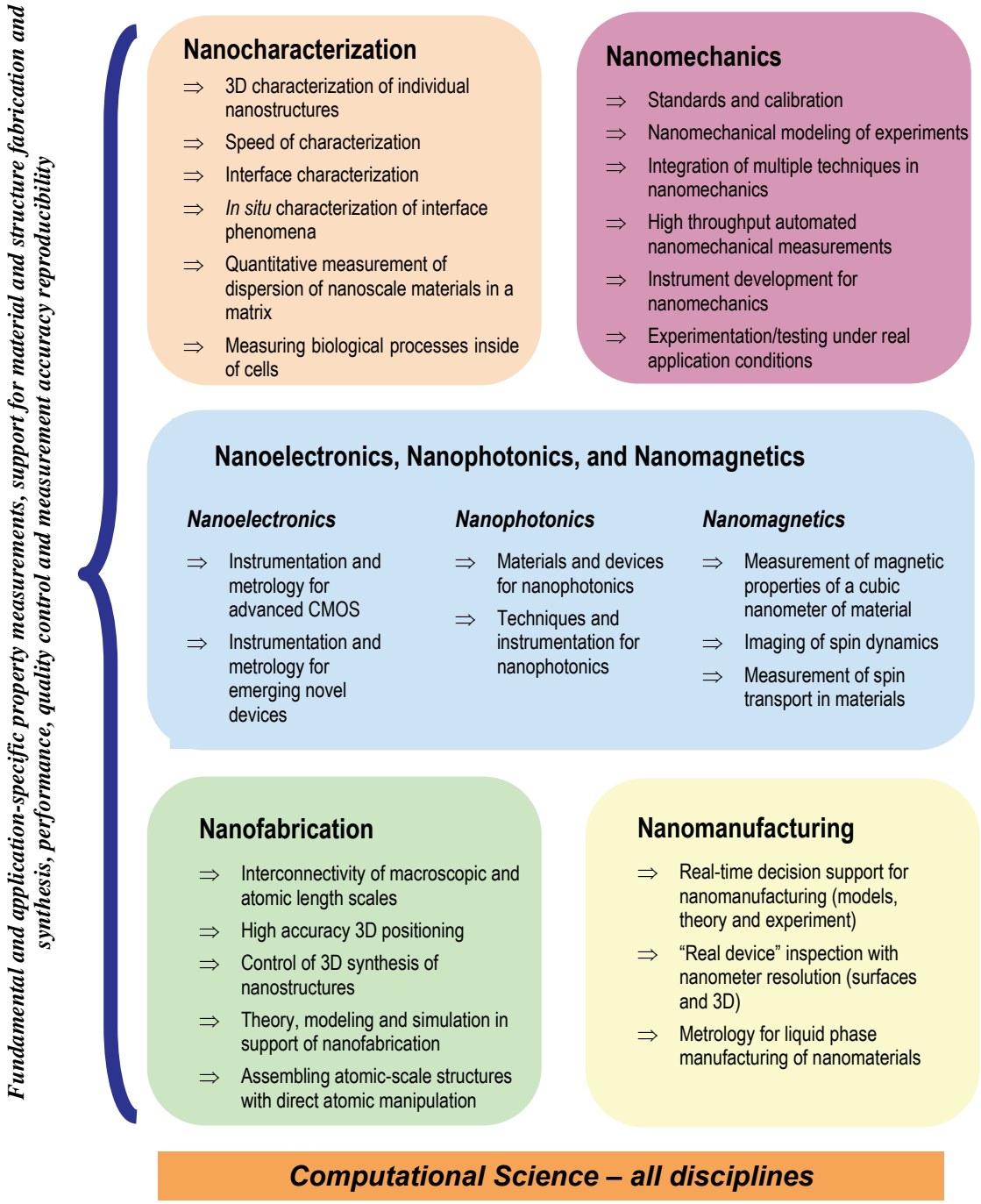

Figure 8.3. Grand challenge topics for nanoscale instrumentation and metrology.

APPENDIX A. WORKSHOP AGENDA

Agenda*
NNI Interagency Workshop
Instrumentation and Metrology for Nanotechnology Grand Challenge Workshop
January 27-29, 2004
National Institute of Standards and Technology, Gaithersburg, MD
Green Auditorium, Administration Building

DAY ONE: TUESDAY, JANUARY 27, 2004

7:00 Registration opens – coffee and pastries available
8:25 Dr. Michael T. Postek, National Institute of Standards and Technology – Call to order

Keynote Session

8:30 Dr. Arden Bement, Jr. Director of the National Institute of Standards and Technology – Welcome
8:45 Hon. Phillip J. Bond, Under Secretary of Commerce for Technology
9:15 Dr. Clayton Teague, Director, National Nanotechnology Coordination Office

9:45 Break

Plenary Session

10:00 Dr. Juri Matisoo, Semiconductor Industry Association – "Grand Challenges as viewed by the Semiconductor Industry"
10:30 Dr. Duncan Stewart, Hewlett Packard – "Metrology for Nanoelectronics and Molecular Electronics"
11:00 Dr. Thomas A. Cellucci, ZYVEX – "Nanotechnology Tools: Solving Real-World Needs"
11:30 Dr. Joergen Garnaes, Danish Institute of Fundamental Metrology – "Needs for Nanometrology from the European Standpoint"

Reports from other NNI Grand Challenge Workshops on Metrology and Instrumentation Issues

12:00 Dr. Barbara Karn, Environmental Protection Agency – Environmental
12:10 Dr. Cliff Lau, Department of Defense – CBRE
12:20 Dr. Barbara Baird, Cornell University – NanoBiotechnology
12:30 Dr. Robert Hull, University of Virginia – NanoMaterials

Introduction to Breakouts

12:40 Breakout session instructions – Joan Pellegrino, Energetics, Inc.

Breakout Sessions Tracks 1 - 5: Current State of the Art

12:50 Working Lunch

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* This was the published agenda as of Jan. 26, 2004. In actuality, the opening of the meeting was postponed until 10 a.m. on Jan. 27 due to inclement weather.

1:15 Plenary Talks
2:15 Organized Brainstorming on State of the Art (Breakouts A1-A5)
3:45 Stretch Break
3:55 Continue Brainstorming
5:00 Assignments for Day 2

5:30-7:00 Reception (Advanced Measurement Laboratory Foyer – enter through the west side of the basement of Building 220 (Metrology)

DAY TWO: WEDNESDAY, JANUARY 28, 2004

7:00 Registration, Coffee and Pastries
8:25 Dr. Michael T. Postek – Call to Order

Plenary Session

8:30 Dr. William Phillips, National Institute of Standards and Technology – "Quantum Computing with Atoms in Optical Nanostructures"
9:00 Dr. Lucas Novotony, University of Rochester – "Instrumentation for Nanoscale Characterization"
9:30 Eric Steel, National Institute of Standards and Technology – "Nanoscale Chemical Characterization: Moving to 3D"

10:00 Break

Breakout Sessions: Future Needs, Barriers and Grand Challenges

10:30 Plenary talks (Future Needs)
11:00 Organized Brainstorming and Prioritization of Future Needs (Breakouts B1-B5)
1:00 Working Lunch
1:15 Gap Analysis/Challenges (Breakouts C1-C5) Organized Brainstorming on Technical Barriers
2:30 Identification of Instrumentation and Metrology Grand Challenges to Overcome Barriers
3:00 Stretch Break
3:15 Completion of Matrices for Grand Challenges
4:45 Preparation for Summary Presentations
5:00 Instructions and Assignments for Day 3

5:30 – 7:00 Reception (NIST Lunch Club and Employee Lounge)

DAY THREE: THURSDAY, JANUARY 29, 2004

7:00 Registration, Coffee and Pastries
8:25 Dr. Michael T. Postek – Call to order
8:30 Review of Combined Breakouts 1A, B, C
8:45 Review of Combined Breakouts 2A, B, C
9:00 Review of Combined Breakouts 3A, B, C
9:15 Review of Combined Breakouts 4A, B, C
9:30 Review of Combined Breakouts 5A, B, C
9:45 Discussion, Holes, Questions and Summary Charge
10:30 Break
11:00 Report Preparation (detailed outline) Tracks 1-5
11:00 Track 6 Breakout Session: Crosscut – Computational Science Issues
Plenary Talks
Brainstorm Barriers and Needs

1:00 Lunch (working) All tracks
1:00 Track 6 Develop Recommendations
2:00 Complete Report Preparation Tracks 1-5
2:00 Complete Recommendations Track 6
2:15 Discuss Next Steps

2:20 Adjourn

DESCRIPTIONS OF BREAKOUT TRACKS

Track 1 – Instrumentation and Metrology for Nanocharacterization.

Co-chairs: Richard Cavanagh (NIST), T. J. Mountziaris (NSF), Nora Savage (EPA), and Hongda Chen (USDA)

Metrology challenges for nanocharacterization span issues in physical and chemical metrology including magnetism, force, hardness, strength, and length measurements, chemical composition determination, shapes of pores and particles, and 3D relationships of complex nanoscale components. Future advances in nanotechnology will hinge on the development of appropriate measurement expertise, the realization of nanoscale 3D imaging capabilities, the acquisition of measurement methods that are scientifically sound and artifact free, and the availability of quantitative metrology that affords analytical capabilities that parallel what is currently achievable on the microscale. To realize these nanocharacterization challenges, a combination of new measurement capabilities will be required that either extend existing measurement techniques such as those used by microscopists and spectroscopists, or emerge from the invention of new measurement methods that enable both compositional and performance factors to be quantitatively and reproducibly measured on the nanoscale.

Track 2 – Instrumentation and Metrology for Nanomechanics

Co-chairs: Clare Allocca (NIST), Kristin Bennett (DOE), Douglas Smith (NIST), Jorn Larsen-Basse (NSF), and Stephen Hsu (NIST)

Nanomechanics refers to the measurement science of the mechanical properties of nanostructured materials or other materials at the nanoscale. These properties include elasticity, hardness, friction, adhesion, and durability. Common tools include scanning probe microscopies (SPM), nanoindenters, and nanotribometers. This track will examine the current state-of-the-art and identify the gaps to reach accurate and traceable measurements of future devices and systems—discussions will cover both measurement needs and instrumentation. A technical roadmap of how some of the uncertainties in measurements may be resolved through instrumentation, practice, calibration, and new measurement tools will be developed.

Track 3 – Instrumentation and Metrology for Nanoelectronics, Photonics, and Magnetics

Co-chairs: Robert Shull (NIST), David Wollman (NIST), Altaf H. (Tof) Carim (DOE), and David Seiler (NIST)

New nanoscale devices and structures are expected to revolutionize the fields of nanoelectronics, photonics and magnetics. Realizing the impact of these advances will require accelerated development of the underlying metrology and instrumentation needed to make reliable, reproducible measurements of device performance and materials properties and to successfully incorporate devices into commercial products. This breakout session will focus on identifying state-of-the-art measurement capabilities in the areas of nanoelectronics, photonics and magnetics

device properties and identifying drivers for developing new metrologies and improving commercial instrumentation. Relevant technologies for this session include: advanced semiconductor devices; nanowires, molecular electronics and other "beyond CMOS" technologies; quantum computing/cryptography; quantum dots, photonic crystals, and optoelectronic nanowire structures; nanoengineered magnetic sensors, magnetic storage and media; spin electronics; etc.

Track 4 – Instrumentation and Metrology for Nanofabrication

Co-chairs: Charles Clark (NIST), Richard Silver (NIST), and Guebre Tessema (NSF)

Nanofabrication involves methods of fabricating device-like structures with features having lateral dimensions down to a single atom. Much of this work is currently focused on fabricating features that are not necessarily accessible to the macroscopic world. Techniques such as modification of hydrogen terminated surfaces and directed alteration of self-assembled monolayers are being widely explored as they are often stable in an ambient environment. There is also a significant focus on the individual manipulation and placement of atoms and molecules. Some of this takes place in a UHV environment, and future work will involve developing methods to interact with these structures and devices with external instrumentation. This session will focus on the broad question of which technologies are most promising, where additional research should be focused, and identifying key challenges in the change from research and development to a manufacturable technology. Other key issues such as the interconnect challenges will be discussed.

Track 5 – Instrumentation and Metrology for Nanomanufacturing

Co-chairs: Kevin Lyons (NIST), Julie Chen, (NSF), James Whetstone (NIST), Xiang Zhang (UCLA), Marylyn Bennett (Texas Instruments), James Arnold (NASA), and Avram Bar-Cohen (University of Maryland)

As nanometer scale product concepts transition to manufacturing, metrology needs will change accordingly. To meet these challenging production requirements, industry will need new metrology tools of various types and quantities. It can be expected that the tool technologies will include most of the current metrology technologies such as near-field optics, scanning microscopy, spectroscopy, and interferometry, although the form factor will likely be different. Equally significant will be the development of new tools designed specifically for mass production applications. Industry will look for metrology tools that do not require UHV environments or stringent vibration isolation, can be configured in mass arrays, support extremely fast measurements, occupy limited production floor space, allow suitable manufacturing work volume, and can be purchased at reasonable costs. Data from the tools must be received in real-time allowing for fast analysis and transformation into information and knowledge. The tools must also provide for rapid set-up (calibration), support reconfiguration for other uses, and support use by manufacturing personnel. These are demanding requirements regarding precision and throughput yet essential for transitioning the nano-product from prototype to production status. An outcome of this session will be a list of promising technologies that can meet the metrology needs for production along with a roadmap that captures the progression of research milestones required to transition these technologies to production.

Crosscut – Computational Science Issues

Co-chairs: Ronald Boisvert (NIST), Alan Karr (National Institute of Statistical Sciences), Mark Lundstrom (Purdue University), and Nell Sedransk (NIST)

The development and utilization of nanoscale instrumentation and metrology will require significant new resources from the domain of computational science. Theoretical and

computational modeling of nanoscale systems provides necessary understanding for the development and improvement of nanoscale instrumentation. As in all measurement systems, understanding the uncertainty associated with such measurement devices can present difficult statistical issues; also, nanoscale measurements also often differ in kind from conventional measurements and require specific new methodology. In some cases, virtual measurement systems, i.e., those based on computational simulation, may be a viable means of performing nanoscale measurements. Modeling and simulation of nanoscale systems is a challenging enterprise, requiring new theoretical, mathematical, statistical, and computational tools, including techniques for the efficient exploitation of high-end computing resources. Finally, with nanoscale instrumentation in place one faces a new dilemma: nanoscale observations leading to terascale databases. The management and use of such data will strain existing tools from computer science and statistics. Visualization may be indispensable for the understanding of nanoscale data. In this crosscut we will attempt to identify key mathematical, statistical, and computational technologies and tools which will need advancement to support the development and exploitation of nanoscale instrumentation and metrology.

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CHALLENGES FOR METROLOGY AND NANOTECHNOLOGY

Donald R. Baer, Pacific Northwest National Laboratory

Opportunity and challenge: Although some areas of nanotechnology, such as the semiconductor industry, have current and well-defined near-term measurement needs, many areas of nanotechnology, and the related metrology needs, are much less developed. At this time it is not possible to clearly identify the long-term instrumental and metrological needs. Nonetheless, it is essential to begin to establish the framework and approach to meet long-term needs and to address areas identified as important.

Nomenclature: There are many different things that we call nanoscience and nanotechnology for a variety of different reasons. These cover different disciplines and a wide variety of technologies as well as different physical, chemical, and biological phenomena. Well-thought-out terminology is an essential element for identification and clarification of the concepts associated with nanoscience and nanotechnology. It is also a critical component in the development of accurate, reproducible and meaningful metrology.

Nanoparticle characterization: Challenges with accurate and useful characterization of nanoparticles highlight a few topics of importance. Several different methods are used to measure and report the *size and size distribution* of nanoparticles. Well-defined and generally accepted standard methods will be essential if nanoparticles are to be widely used and distributed as "industrial" materials, particularly for size-dependent applications. However, the information needs for nanoparticles do not even begin to be satisfied by knowing the size. Any useful reporting of nanoparticle properties must include information related to *production process, time after production, environmental conditions* and *handling history*. There are also complications associated with the *collection of nanoparticles* for analysis or storage and the *movement or transport* of nanoparticles from one environment to another for analysis (or use) without contamination or unwanted alteration.

Although traditional measurements of structure such as transmission electron microscopy (TEM) and X-ray diffraction (XRD) are critically important, it appears that they do not always provide an adequate picture of the detailed structures of nanoparticles. Synchrotron-based small-angle scattering measurements provide an indication of structure not apparent in the TEM or XRD data. Contamination and surface structure will play a major role for chemical applications of nanoparticles and our ability to accurately obtain such information on very small particle surfaces is very limited. For nanostructured materials, it may be essential to interactively *combine multiple types of data* to enhance the information obtained.

Distributions, statistics and other challenges: As already noted for nanoparticles, it is essential to have an accurate sense of the structure and size distribution of the components of nanostructured materials. This is an immediate issue for current and near-future generations of integrated circuits. Time-resolved single-molecule spectra show a significant variation with time. The relative magnitudes of various spectral components reflect many factors including flexing of the molecular structure. Although the time-averaged signal is identical to the spectrum of bulk material, a series of time-resolved single-molecule spectra contain significant additional information. In some situations it may be that the statistical distribution of data as collected can be analyzed to provide information about the distributions, sizes, motions, and even functions of nanostructured materials.

In most cases nanosized components will need to be integrated into larger meso- to macro-sized systems. There are significant challenges in designing, assembling (including registry and electrical contacts), and verifying the nature of the structures achieved. In addition to contamination issues, there are issues related to the stability of nanostructures when exposed to photon or particle irradiation. Methods of confirming the robustness of the final material and functional properties will need to be developed.

REPORT ON THE NSF/NIH/NNI WORKSHOP ON NANOBIOTECHNOLOGY HELD OCTOBER 9–11, 2003

Barbara Baird, Cornell University

Substantial segments of the scientific community are confident that nanoscience and nanotechnology (NT) will revolutionize research on biology and medicine. Yet, much of the biomedical research community has at best passing familiarity with the novel relevant discoveries that are emanating from the larger NT communities, ranging from physics, to chemistry, to engineering and biosciences. On the other hand, nanotechnologists are developing new tools, but they often have a limited understanding of the needs of the biomedical communities, or of the restrictions that biology (and medicine in particular) place on the proper design of nanotools or nanosystems. The purpose of this workshop was to convene thought leaders in biomedical and NT research and to identify cross-cutting scientific opportunities that can be realized only through effective collaboration among these communities. The workshop was co-chaired by Viola Vogel and Barbara Baird, with a steering committee that included representatives from NSF, NIH, and NNCO. This presentation will focus on metrology issues. A written report on the entire workshop is in preparation [now complete: http://nano.gov/nni_nanobiotechnology_rpt.pdf].

NANOSCALE MECHANICAL PROPERTY MEASUREMENTS

Shefford P. Baker, Cornell University

The NNI seeks to realize new technologies based on materials, properties, and functional densities that become available when the structure of a material or dimensions of an object are controlled at nanometer length scales. As we continue to improve our ability to manipulate and control materials at smaller length scales, and apply these methods to new materials ("soft-" and "bio-"materials), powerful new technologies can be expected.

One of the fundamental requirements for any engineered device, regardless of whether its primary function is electrical, mechanical, optical, chemical, magnetic, or biological, is mechanical stability. In short, devices must retain mechanical integrity to remain useful. Thus, understanding the mechanical properties of nanofabricated materials and objects will be critical to understanding and improving the reliability of nanofabricated devices. However, two aspects of the nanoscale conspire to make this difficult. First, the mechanical properties of materials often deviate from bulk scaling laws as key dimensions become small, and furthermore, many materials exist only on this length scale (e.g., carbon nanotubes, protein molecules). Thus mechanical properties must be measured at the nanoscale. Second, however, such measurements are difficult. For example, one must impose and measure displacements that are some orders of magnitude smaller than the object being measured. Further complications are provided by the fact that nanofabricated devices are typically composites of dissimilar materials, and that new materials, particularly soft materials, are being used. Three areas in which progress in metrology must be made include the following:

Dynamic mechanical nanocontact measurements and analysis: both depth-sensing indentation (DSI, also known as nanoindentation) and scanning force microscopy (SFM) methods have been developed for nanomechanical measurements. However, to date, accurate, quantitative mechanical property measurements can only be reliably obtained from quasi-static measurements of linear elastic materials using DSI. As more and more "soft" materials (e.g., polymers, biomaterials) are developed and incorporated into nanodevices, it will become more and more important to be able to obtain quantitative information about dynamic properties, such as loss modulus, damping, and viscoplasticity [1-3]. This will require both the development of testing equipment and methods (the dynamic behavior of the testing machine must be very well understood) and analysis models.

Stress measurement methods for nano-objects: because of thermal expansion mismatch, lattice mismatch, and volume changes resulting from microstructural evolution, nanofabricated devices typically experience very high stresses, which may affect the performance of the part, and can result in failure of the device because of deformation or fracture. To understand the mechanical behavior of such devices, it is necessary to interrogate the internal structures to determine stress levels and, if possible, deformation. This can be accomplished without the need to section the sample (thereby creating artificial free surfaces) using energetic beam methods. For example, synchrotron X-ray diffraction methods have been used to determine strains and stresses in differently oriented grains in thin metal films [4, 5]. Metrology needs for this work include development of smaller high-intensity beams, specialized sample stages, faster goniometers with access to a wide range of geometries, and methods for interrogating amorphous materials.

Adhesion and interface phenomena: nanofabricated devices typically include a high density of interfaces with a wide variety of materials combinations that affect mechanical behavior in a number of ways. One of the most obvious is deadhesion [6, 7]. Interfaces can also determine how much load is transferred to an individual nanocomponent, the rate of diffusion (and accompanying deformation) in the material, crack propagation (or deflection), and constraints on plasticity. Interface contributions to mechanical behavior are only poorly understood and represent one of the areas where detailed study at the nanoscale would pay off.

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LENGTH, MASK AND ANGLE METROLOGY

H. Bosse, Physikalisch-Technische Bundesanstalt (PTB)

Since 2001, I have been responsible for the German National Metrology Institute Department of Length and Angle Graduation Metrology. We are dealing with one-dimensional (1D) length metrology on precision graduations with length up to 600 mm, with photomask and wafer metrology on objects up to 300 mm × 300 mm and with angle metrology. The measurement uncertainties are in the range of a few nanometers or nanoradians in the most challenging applications. To cope with these requirements, specific reference instrumentation as well as suitable modeling of the measurement process is indispensable. The instrumentation we currently use in our department for the most challenging applications is as follows:

1D length metrology: nanometer comparator: 1D vacuum comparator with 610 mm travel range, and calibration of line scales and incremental length encoders or other type of grating-based systems, targeted uncertainty is < 5 nm.

Mask metrology: 2D optical mask comparator with 230 × 230 mm travel range, low-voltage SEM metrology system with 300 × 300 mm travel range. Equipment is used for length and registration metrology as well as CD metrology, mainly on masks but also on nanostandards, like 1D and 2D precision gratings, uncertainties targeted at < 10 nm for registration and CD and < 10 pm for mean pitch.

Angle metrology: High-precision angle comparator for calibration of measuring devices for 360 degrees as well as small angles; angle positioning uncertainties 0.005 arcsec or 25 nanoradian (25 nm @ 1 m radius).

Requirements for calibration are often twofold: absolute length measurement results, traceable to the SI meter definition, and high-precision determination of deviations from an ideal graduation. The latter requirement is a result of the application of length and angle measurement systems used in production control. To achieve the smallest possible uncertainties for length measurements the relative displacement between sample and measurement system should be realized to allow a direct traceability to the SI definition of the meter; that is, the displacement should be measured by *vacuum interferometry* (or wavelength compensating interferometry in air) on the basis of one of the recommended wavelengths. Knowledge of length compressibility of the sample as well as the thermal expansion behavior is also crucial for length metrology on larger objects with uncertainties targeted in the nanometer regime. Some thoughts on future needs in dimensional nanometrology are briefly stated.

Short-Term Needs

- Improvement of probe sample interaction modeling, refinement of models by measurements with improved reproducibility on better defined reference samples with better-known material parameters
- Closer cooperation with high-end manufacturing sites to jointly develop better standards that then can be calibrated with smaller uncertainties
- Improvement of cross-calibration procedures between different metrology instrumentation
- Evaluation of (micro-) scatterometry methods for in-line production monitoring, comparison with other high-resolution microscopy
- Improvements of high-quality graduations (profile line shape, smaller pitches)
- Improvement of atomic force microscope (AFM) measurement speed
- Measurands to take into account 3D shape of isolated or grouped structures

Medium- and Long-Term Needs

- Combination of different probing systems in one instrument to improve direct comparability of measurement results
- Combination of stable self-assembled structures (locally perfect) with globally defined high-quality graduations

- Increase potential of X-ray interferometry for dimensional metrology (speed!)
- Redundant measurement techniques in high-precision dimensional nanometrology (e.g., trilateration, error separation)

CHARACTERIZATION OF NANOPARTICLES IN TISSUES

Stanley A. Brown, FDA/CDRH/OST

There is a need to develop an understanding of the interactions between nanoparticles and human tissues. These particles may be introduced by respiration or ingestion from the environment, percutaneously as with sun screens, orally or systemically as medication or diagnostic media, or directly *in vivo* as wear and degradation products from implants. Of particular interest to the FDA Center for Devices and Radiological Health is the release of particles from implants. It is becoming clear that wear debris from "hard on hard" total hip prostheses bearing surfaces of metal-on-metal, and ceramic-on-ceramic combinations of cobalt chromium alloy or alumina produce particles in the 10–50 nm range. Degradation of nanocomposites of bioactive ceramics such as hydroxylapatite or glasses may also be in the nanometer range. It is unknown whether other resorbables materials and tissue-engineering matrix materials will produce nanoscale particles. It is also unknown whether cellular enzymatic and oxidative degradation mechanisms used for wound healing, bone remodeling, and combating infections may also break down particulate debris into the nanoparticle size.

To gain this understanding about cellular effects of nanoparticles, we need to develop the methodologies for identifying, isolating, capturing, and characterizing particles. First we must identify where in organs or tissues the particles may be located. Then we must isolate those regions and capture the nanoparticles for characterization. Characterization at a minimum must establish particle size, morphology, composition and surface chemistry. It is imperative that we can validate that these protocols have no effect on the surface chemistry and morphology of the particles. This challenge is confounded by that the fact that the effects of the particles may not be caused by the particles themselves, but may be a reaction to the particle–protein complexes formed *in vivo*. Thus there may be a need to characterize both the particle and the complex.

CALIBRATING AFM PROBES

N. A. Burnham, Worcester Polytechnic Institute

Much of my previous work has focused on the interpretation of nanomechanical data gathered by means of atomic force microscope (AFM) because I wanted to have a general idea of what the data were revealing. Now, with a good general understanding of my data, I have turned to trying to improve the quantitative aspects (metrology!) of the instrumentation.

Our group has recently published a comparison of cantilever calibration methods for the spring constant of AFM cantilevers [1]. A manuscript on a simple radius calibration method for spheres attached to AFM cantilevers has been revised for Review of Scientific Instruments [2]. The technique could be extended to parabolic or conical shapes as well. We are working on extending the work in [1] to stiffer cantilevers and addressing the precision and accuracy of the stiffness calibration [3].

Depending on the supplier, it is now possible to buy AFMs with good closed-loop detection or other means for knowing the real position of the scanner. My next concern after the calibration of the spring constant of the cantilever and the radius of the tip have been adequately treated will be the wear and material-transfer properties of the tip. Here a nonreactive, durable, stiff material where the surface chemistry remains stable would be a good choice.

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CHALLENGES IN NANOSTRUCTURE SYNTHESIS

Alex de Lozanne, University of Texas at Austin

The current challenges in nanofabrication synthesis can be broadly divided into two areas: invention/discovery and manufacturing. The possible solutions to the challenges in one of these areas are likely to be quite different from those in the other area because of the dramatic difference in the desired throughput and the initial investment required. Most of us who have worked with scanning probe techniques for a long time believe (and many of us have practiced) that such techniques are the best for invention/discovery in the nanoworld. Some would go as far as trying to extend scanning probe techniques to the manufacture of nanotechnology—a worthwhile quest, yet one that is less likely to become practical. I shall therefore restrict my comments to invention/discovery.

The more the merrier: When it comes to inventing or perfecting nanodevices, or discovering interesting effects that may be technologically useful, serendipity plays a big role. For this reason it would be wise to make nanofabrication tools more widely available, chiefly by lowering the price of the technology to the point that every student in science and engineering can have access to such tools. If we set a maximum price tag at $10K, the AFM is perhaps the only nanoscale fabrication/characterization tool that can fit on such a budget. At this time it would be hard to imagine that a company could survive by selling such a low-cost instrument. However, I believe that one or two of the established groups in the country could be funded to develop detailed plans and instructions to allow others to build such an instrument for less than $10K. Later this could develop into a low-overhead commercial venture.

Tips: Similarly, the cost of cantilevers/tips needs to come down by a factor of 10, namely to the order of $1 a piece. *In situ*-grown carbon nanotube tips are perhaps the most promising technology to reach this price range. Although our group and others have developed methods for growing nanotubes directly on SPM tips, so far the yield is not high enough to attempt mass production, or the process cannot be scaled up. Again, it would be wise to fund a research group with the explicit goal of finding a process that will mass-produce AFM tips in this price range. Tips that have more robust mechanical properties than carbon nanotubes would also be desirable for the more "brute force" approaches to nanofabrication.

Other benefits: Although having many thousands of college students playing with an AFM may not produce any new devices, it will at least increase the level of education regarding nanoscience/technology. Furthermore, it would be a boost to all research groups working with SPM because it would increase the pool of young students with hands-on experience—and hopefully interest—in SPM.

Making the basic technology of AFM more accessible would also allow research groups to use it to build more sophisticated instruments, for example, for operation in vacuum or low temperatures. This would allow for those of us using more powerful methods such as STM-CVD or atom manipulation to benefit from the savings. This is not a trivial benefit, as even in well funded groups the progress of a student may be slowed down by shared access to SPM tools.

NANOTECHNOLOGY-BASED METROLOGY NEEDS: IDEAS FROM THE SEMICONDUCTOR INDUSTRY

Alain C. Diebold, SEMATECH

In 2004, the integrated circuit (IC) already contained millions of devices that can be classified as nanotechnology. The feature dimensions of transistors in microprocessors have gate lengths less than 40 nm and the *International Technology Roadmap for Semiconductors predicts gate lengths of less than 10 nm by 2015* [1]. The dimensions of gate dielectric layers in today's transistors are less than 2 nm in thickness, and

on-chip interconnect wires and the vias that connect layers of these metal wires have barrier layers close to these dimensions. The interfacial layers between these thin films are engineered during the fabrication of these transistors. Successful manufacturing of ICs requires metrology that controls the multimillions of transistors on each chip. Defects that serve as yield killers have similar dimensions to the features themselves. It is important to note that the manufacturing processes and equipment that will be used 3 years from now in the 65 nm IC technology node (with microprocessors having 25 nm transistor gate lengths) are already being tested by IC manufacturers. The progression of alpha tool to beta tool to final tool takes 1 year for each step. Thus, the physical and electrical measurement technology for this technology node is also being tested. *The development and manufacture of these nano-ICs requires that materials characterization and metrology develop ahead of the process tool development if we are going to be ready for measurement needs 10 years or more in the future. A critical aspect of this development is the availability of features fabricated from the materials that will be used in the future.*

A key concept for manufacturing process control is the need to rapidly collect statistically significant data. Today, we often measure one value from a local distribution of values (one site on a wafer) or an average value for that local distribution. Ideally, we would measure the average and width of the local distribution in a way that reflects the variation across the chip. Inherent is the growing concern that test structures do not reflect actual on-chip variation. Another implicit concept is that measurement on patterned structures is critical. So-called out-of-the-box ideas such as measuring critical dimensions (e.g., transistor gate length) using a method that never actually images the individual features must be considered. The example here is the work of Ausschnitt [2]. Instead of measuring the width of individual lines, the average length of an array of lines and spaces is measured. This information is then used to control the focus and exposure of the lithographic patterning step. The final result is control of the critical dimension of line width. *The ability to control average transistor line width and reduce the range of values will become more important as well as more difficult.*

Optical and electrical measurement methods have a long history of moving from lab to the FAB (IC factory). Interface sensitive methods such as optical second harmonic generation have been underused. Thanks to the availability of new laser technology, ultra fast optical methods can be applied to the materials of the future. Faraday measurements can be applied to spin transport in spintronics. X-ray reflectivity provides the ability to measure and control buried interfaces in opaque materials. Its application to patterned features and future materials stacks requires much closer ties between the laboratory systems and the clean-room-compatible systems that use optics capable of rapid measurement. Once again the need to couple metrology research and development to the materials set needs to be a mandatory aspect of every project. Although the devices of the future are predicted to be rather similar to those used in today's IC, the use of new substrate materials such as silicon on insulator, strained silicon on insulator, or germanium on insulator greatly impact the measurement itself. New transistor device structures such as the so-called FinFET may change the orientation of the film thickness measurement from horizontal to vertical [3].

It is important to note that the ITRS already contains sections on emerging device technology and the integration of high-frequency communications capability. There are real metrology needs in each area.[1]

In addition to the individual process measurements, the need for development of new microscopy should not be overlooked. Although microscopes are specialized to meet the needs of specific applications such as line width (critical dimension) measurement, the link between the developments of advanced transmission electron microscopy (TEM) and its subsequent effect on scanning electron microscopy (SEM) deserve mention. Aberration correction lenses for TEM are moving into some applications of SEM. High-voltage SEM is being considered as a potential method for future linewidth measurements. The point projection microscope (electron holography) remains a potential microscope for applications beyond the 10-year horizon. The big advantage is the ability to measure average line width and the width of the distribution of linewidths. *The effect of TEM and TEM microanalysis on materials and process development will continue to be a key method for all nanotechnology. Thus the TEAM (Transmission Electron Aberration—Corrected Microscope) project is a fundamental requirement for future metrology.* The ultimate view of all materials would be an atom-by-atom map of a nanostructure. The local electrode atom probe is being developed and requires an infrastructure of national lab and university experts for method improvement and applications development.

A review of the metrology needs for the IC industry, including a look at those required for nanotechnology, is available in the proceedings of the NIST-sponsored conference on Characterization and Metrology for ULSI Technology [3]. This review is based on the semiconductor industry's ITRS Metrology Roadmap.

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FROM MACRO- TO NANOMANUFACTURING: MAKING THE QUANTUM LEAP HAPPEN

Haris Doumanidis, University of Cyprus

This presentation overviews the philosophy, implementation, and portfolio of the new Nanomanufacturing Program at NSF. This is placed in the context of recent downscaling processing and miniaturization research developments, as well as the National Nanotechnology Initiative and its connection with nanoscale science, engineering, applications, and societal impacts. The program focuses on scale-up synthesis of functional structures, devices, and systems and integration across dimensional scales and multiple energetic domains, as well as biomimetic approaches. This calls for research programs in nanoscale materials, processes, instrumentation, modeling-control, and design/integration of nanostructured products, and related needs and opportunities are identified. Among implementation strategies, emphasis is placed on the role of education and training, interfacing between academic, industrial, government, and professional institutions, outcome dissemination and effect on the society. Current information and funding resources are also reviewed.

INSTRUMENTATION AND METROLOGY FOR NANOFABRICATION

Haris Doumanidis, University of Cypress

State of the Art

Over the past 2 years, the Nanomanufacturing Program at NSF has funded research relating to nanoscale instrumentation and metrology in the following themes:

- FIB micromachining and advanced characterization of carbon nanotube–metal junction (K. Dovidenko, SUNY Albany)
- Staggered probes for integrating nanomachining and metrology (R. Vallance, Univ. of Kentucky)
- Nanofabrication using subwavelength near-field nano-optical laser processing (S. Das, Univ. of Michigan)
- Novel low-cost nanolithography technique using nanometric high-transmission optical antenna (X. Xu, Purdue Univ.)
- Nanoxerography: the use of electrostatic forces to pattern nanoparticles (H. Jacobs, Univ. of Minnesota)
- Nanorobotics (A. Requicha, Univ. of S. California)
- Motion control platform for accurate measurement and manufacturing of nanostructures (S. Smith, Univ. of N. Carolina)
- Advanced control algorithms for active materials actuators used in nanoscale positioning (S. Seelecke, N. Carolina State Univ.)

- Protein-based nanomotors and nanorobots (C. Mavroidis, Rutgers Univ.)
- Manipulation and 3D organization of nanoparticles by dielectrophoresis (P. Alexandridis, SUNY Buffalo)
- Spatially resolved characterization of nanoporous SiC layers (S. Ostapenko, Univ. of S. Florida)
- Torque spectroscopy for nanosystem characterization and fabrication (D. Cole, Duke Univ.)

Research Issues, Barriers and Needs

The following research themes are crucial in further development of instrumentation and metrology for nanofabrication:

- Multienergetic domain transduction and measurement techniques at the nanoscale
- Multiscale integrated instrumentation, from macrosensors to MEMS to NEMS, etc.
- Simultaneous fabrication and in-process sensing integrated in one machine
- Lab-on-a-chip/fab-on-a-chip integrated fabrication/metrology MEMS instruments
- Parallel sensing element arrays and scanned sensing systems for field measurements
- 2D surface mapping and 3D volume imaging sensing technologies (e.g., ultrasonics, etc.)
- Off-line metrology and real-time sensors for feedback control during fabrication
- Improved resolution and bandwidth for distributed, dynamic control
- Decoupling of transduced information in complex nanoscale structure-sensor interactions
- Model-based software observers of unmeasurable nanostructure states
- Positioning, orientation, alignment and registration methods between the structure and sensor
- Redundancy on nanosensor elements for robustness to instrumentation defects
- Power autonomy for activation of measurement instrumentation at the nanoscale
- Multiscale grid interconnects and wireless transduction/interrogation of information
- Scalability and affordability of nanometrology instrumentation for mass manufacturing and use

Future Milestones and Interdisciplinary Breakthroughs

New developments and areas in instrumentation are expected in the longer term:

- Computational virtual nanoenvironments for geometrically coordinated measurements
- Cognitive signal transduction by neuron on silicon and remote action potential research
- Quantum effects for nanostructure/sensor transport, transcription and transduction
- *In situ* hardware feedback control by integrated nanosensors, controllers and actuators
- Bioinstrumentation and metrology, based on biological phenomena for measurement, including biomimetic instruments, bioinspired measurement techniques, hybrid bio/abiotic sensor elements, and use of biological entities for metrology (biomolecules, viruses, etc.)

Supporting Infrastructure

- Education and training in nanoinstrumentation and metrology with various modalities
- Networked laboratory infrastructure for nanometrology (NIST, NNIN, etc.)
- Teleoperation of instruments (telefabrication and telecharacterization) via cyberinfrastructure
- Collaboration with precision engineering/metrology societies and international centers

FUTURE NEEDS FOR NANOMETROLOGY AND GRAND CHALLENGES FOR RESPONSIBLE COMMERCIALIZATION OF NANOTECHNOLOGY

G. S. Blackman, K. C. Doraiswamy, S. C. Freilich, W. D. Provine, A. H. Reid, J. C. Romine, D. Scott, S. Subramoney, and D. B. Warheit, DuPont

Nanotechnology is already here and has been for decades! DuPont has been manufacturing products with nanoscale architecture for a long time. We make polymers in which the unique mechanical properties depend on 10 nm hard segments buried in a sea of liquid such as polymer chains. Films only a few molecules thick protect fabrics and molded and painted parts from stains and the environment. Recently there has been a dramatic improvement in our ability to measure, characterize, visualize, and manipulate matter on scales that could only be imagined a decade ago. However, there are still significant challenges in metrology and characterization that if not overcome will hamper our ability to discover and commercialize new nanostructured materials, nanoparticles, and nanocomposites. The challenges below address specifically our interest in nanoparticulate systems.

Grand Challenges and Hard Problems

Determine coverage, thickness, and uniformity of thin coatings on nanoparticles: Nanoparticles are almost always coated with something. In their natural state there is so much surface area, so many unsatisfied surface states, that unless we do something to stop it they will agglomerate to reduce the overall surface free energy. Coatings can be organic dispersants or surfactants, or thin inorganic coatings. In the final article the surface coating can provide reactive sites to tie the nanoparticle into the polymer matrix to improve mechanical or other properties. Failure to engineer this surface appropriately can actually weaken the material instead of toughening it. Developing and applying the functional coatings is difficult enough, but determining the coverage and perfection of nanometer-thick surface coatings on nanoparticles presents a significant challenge. Any technique with sufficient resolution to analyze a single particle will need particular scrutiny to avoid statistical pitfalls.

Determining the surface chemistry of nanoparticles on the nanoscale: In most cases the maximum benefit of nanomaterials will rely on their successful incorporation into functional structures. Self-assembly of hierarchical structures, via, for example, chemical templates or DNA-directed assembly, is expected to be a valuable part of the manufacturing processes. Present measurement techniques tend to see only the average or distribution of surface properties, not the individual active sites. When the interface of interest is buried or hidden within a matrix the problem becomes even more difficult. A high-resolution means of quantifying the surface chemistry of nanoparticles is needed so that specific molecules can be attached to improve functionality or to construct (or control the assembly of) 3D structures.

Statistical evaluation of dispersion of nanoparticles from synthesis through manufacturing and into the final consumer product: As a result of the natural tendency for nanoparticles to agglomerate to reduce their overall surface area, complete dispersion during all phases of manufacturing, and especially in the final nanocomposite material is of critical importance. If particles do agglomerate during one phase of the synthesis or manufacturing process, it is extremely challenging to get them to redisperse. Techniques exist to evaluate particle size and distribution in solutions and in free-flowing powders, but the quality of the data depends on our ability to ensure complete disaggregation. When the particles are compounded into a solid matrix it is much more difficult to get a reliable, statistically significant measure of the dispersion. There are also challenges in measuring particle size distribution as the particles get smaller or when the particles are anisotropic in shape (nanotubes, nanorods, exfoliated clays). Of particular interest are methods to evaluate the length distribution of collections of anisotropic materials such as carbon nanotubes.

Predicting macro/bulk or end-use properties based on nanoscale tests: It is difficult to predict the performance of a nanostructured material based on small-scale measurements. The end-use application may be complex, such as integration issues in a thin dielectric film or toughness and durability. Instruments to evaluate nanomechanical performance of polymers exist, but the connection of nanohardness, nanoscratch, and so on to end use is often difficult to establish. In those applications in which the material will be used in thin film form or the mechanical damage is confined to the surface or near-surface region, existing

nanomechanical tests can provide useful information about mechanical performance. However, even in such a case, extracting intrinsic mechanical properties from the measurements is seldom straightforward.

Instrumentation or methods to sort or select nanoparticles based on size or properties: Successful commercialization of a nanostructured material may require the ability to select or sort the nanoparticles before use. For example, we may want only the semiconducting carbon nanotubes or a particular chirality, or we may need to eliminate particles above a certain size because of a deleterious effect on mechanical or optical properties. Technology considered for this challenge will need to be robust, rapid, and suitable for incorporation into a manufacturing environment.

Safety and toxicology of nanomaterials: There is a paucity of safety data on health risks related to exposures to nanoparticles. The major routes of potential occupational exposures to nanoscale materials are through the respiratory tract (inhalation), the skin, and the gastrointestinal tract (via oral or inhalation exposures). Two of the major metrology-related challenges are precise particle size measurements while generating nanomaterial aerosols for inhalation toxicity studies, and assessment of particle deposition patterns in the lungs of experimental animals. DuPont is committed to the safe and environmentally responsible introduction and commercialization of new nanomaterials that will benefit society, and it is dedicated to understanding the effect of nanomaterials in both their benefits and any potential risks to safety, health, and the environment. DuPont will support the development of standards, methods, and instrumentation to enable the safe development and use of nanomaterials.

APPLICATION-DRIVEN NANOMECHANICS INSTRUMENTATION AND METROLOGY FOR NANOMECHANICS

Craig R. Friedrich, Michigan Technological University

Nanotechnology and nanoengineering represent two diverse entities of the same endeavor. That endeavor is to study and manipulate atoms and molecules to improve products. Nanotechnology generally encompasses the study of nanoscale phenomena whereas nanoengineering implies the design and creation of new or improved materials. Knowledge of the properties of such materials, and the performance of the systems and products they will improve, is necessary to ensure safety, reliability, and economic gain.

The strength of single tubes, although important from a scientific viewpoint, may not be of primary importance in many applications. However, it is conceivable that carbon nanotubes will find acceptance in electronic and thermal applications where the strength, for example, is important but of secondary interest. In other areas, protein-based sensors will likely be integrated with single-electron transistors, or molecular electronic circuits will likely be integrated into stabilized nanofilms in application scenarios. Therefore, measuring the mechanical properties of nanoengineered materials must not be addressed as an isolated endeavor but, rather, integrated with the operating environment and function of the material.

The dominant instruments used to measure mechanical properties such as strength, elasticity, and hardness are probe microscopes and nanoindentors. A survey of the recent literature just on the measurement of the Young's modulus of single-walled carbon nanotubes illustrates the need for standardized techniques and instruments. One experimental genre uses one fixed and one movable probe microscope cantilever to measure force and displacement. However, the influence of the stiffness of the cantilever tips relative to the effective stiffness of the nanotube itself has largely not been addressed. Another approach uses the bending of vertical forests of nanotubes to deduce the effective bending stiffness EI. A nanoindentor tip is pushed into the forest, causing single-walled nanotubes to deflect laterally resulting in bending of many nanotubes because of lateral "contact" forces. From the dimensions of the nanotubes, I is calculated, and the resulting E is found. This approach does not consider the sliding force of one nanotube along adjacent tubes as they bend, which has been found by studies on nested nanotubes to be significant. Questions can be raised about: (1) the meaning of structural properties, such as I, at the nanoscale where discrete materials are measured rather than continuum materials; (2) how these material properties change if electrical current, for example, is carried by the tubes (is there effective stiffening or weakening?); (3) how, if functional side-groups are added to nanotubes to sense oxygen, for example, these change the mechanical properties; and about other topics.

Instrumentation and Methods

The information above shows the need for specialized instruments that measure mechanical properties of materials under the operational conditions of those materials. This would likely lead to nonstandard instruments that are application specific because probably no single instrument can simulate all environments. Fundamental metrology technologies such as probe microscopes will likely be modularized so a single test bed instrument can be outfitted for a variety of operating environments. Some of these environments will be in vacuum, others within other materials, and yet others within living systems. However, the design, fabrication, characterization, and calibration of those instruments should still fall within standard guidelines. The techniques of measuring and reporting properties should be standardized so there is parity across instruments and researchers. As mechanical properties are coupled with more environmental factors, which of those factors are the independent variables and how they are measured and reported will have increased significance. There may also need to be far more reliance on statistical significance testing as a fundamental data analysis tool to determine the relative effect of coupling factors. These testing algorithms should be transparent to the user and also standardized. One lesson that can be learned from the MEMS era is that standardization of test methods and instruments can lead to more rapid acceptance by technology users and better development tools by technology deliverers. The initial development of new metrology tools will most likely take place by end users instead of instrument suppliers because the instruments will be a technology push instead of a market pull. Therefore, resources must be made available to individuals and groups of researchers, who will undertake the development of these instruments. These resources include funding sources and incentives (individuals), collaboration with other instrument developers (industry), and standardized test beds where instruments and their technologies can be compared (government labs).

NEEDS FOR METROLOGY FROM THE EUROPEAN STANDPOINT

Joergen Garnaes and Kim Carneiro, Danish Fundamental Metrology

This presentation will discuss the needs for nanometrology relative to the position paper "The need for measurement and testing in nanotechnology," compiled by the High Level Expert Group on Measurement and Testing under the European Framework Programme for Research and Development. The presentation aims to identify new needs for research and development in metrology (including both measurement and testing), to support the demands from nanotechnology, which is foreseen to be one of the major new technologies of the coming decades. After a brief introduction to nanotechnology the presentation addresses nanometrology from the following perspectives: written standards, scientific instrumentation, validated measurement procedures, measurement standards, chemical analysis, and biology. It is suggested that despite the multidisciplinary nature of nanoscience and the multisector nature of its industrial applications, nanometrology can focus on a few generic developments. Hence it is suggested that the same measurement standards can support the three different industrial sectors: precision engineering, micro- and opto-electronics, and bio-molecular technology.

COMPUTATIONAL SCIENCE IN NANOMETROLOGY

Sharon C. Glotzer, University of Michigan

Precise, accurate, and reliable measurement capabilities are required in all areas of science and engineering. Achieving such capabilities at the nanoscale is especially challenging because of uncertainties and noise inherent to the measurement process at this scale. However, designing, fabricating, and manipulating nanoscale components to make materials and devices will require precise, accurate, and reliable measurements of

- *Forces between nanoscale components:* For example, between quantum dots in organic and aqueous solution for nanosensors; nanotubes and organic molecules on silicon or gold surfaces for nanoelectronics; biological ligands and nanocrystalline surfaces for nanobiomaterials, etc.
- *Nanoscale features of structures fabricated at the nanoscale or via nanoscale components:* For example, cylindrical or lamellae structures formed by the self-assembly of functional nano building blocks; porosity, domain size, positional and orientational arrangement of polymer-tethered, inorganic silica

"nanocubes" used for coatings or low-k dielectric materials; dispersion of nanoparticles in polymer matrices and structure of polymers near nanoparticle surfaces, and so on.

- *Time-dependent processes occurring at length scales ranging from angstroms to micrometers:* For example, dynamics of assembly as molecules and nano building blocks organize into structures with positional or orientational order on increasingly larger length scales.

In all aspects of nanoscale measurement, reliable models based on sound, scientific theories will be needed to interpret measurements, and simulations will be needed to test these theories, validate measurements, and stand in for measurements not yet possible. Computational science has a critical role to play in nanometrology in (1) aiding the interpretation of measurements at the nanoscale, (2) providing fundamental information and insight for the development of nanometric tools and methodologies, (3) providing virtual measurements of nanoscale features or properties, and (4) enabling fast, global access to real and virtual nanoscale measurements. The field of computational science is already contributing to revolutionary advances in nanoscale science and engineering. In nanoscale metrology and instrumentation, major breakthroughs and improvements in many key areas of computational science, mathematics and statistics will be required over the next decade. For example:

- *Order(N) methods* to enable *ab initio* calculations on larger numbers of atoms than is currently possible are required for prediction of, for example, effective forces, structure, and optical, electronic, and mechanical properties as virtual measurements or to validate experiment.
- *Acceleration algorithms* are needed for significantly advancing timescales of quantum and classical simulations for virtual measurements of electron transport, self-assembly of nano building blocks, and electronic, optical, photonic, mechanical and other properties.
- Increased accuracy, extensibility, and transferability of *classical force fields* are needed to simulate (e.g., the assembly of nanomaterials and interpret measurements of structural evolution). Here there are particular challenges in developing accurate force fields for *hybrid materials* such as biological–inorganic and organic–inorganic nanocomposites.
- Improved, public domain *data-mining and database management tools* are needed for accessing/sharing real and virtual measurements.
- Public-domain *statistical analysis tools* are needed for interpreting/assessing *uncertainty and statistical correlations*.
- Faster algorithms and hardware are needed for visualization, especially *interactive visualization* of nanoscale structure and phenomena and *computational steering* of virtual measurement methodologies.
- Theoretical and mathematical methods such as *homogenization* for *coarse-graining* are required for the development of computationally tractable models used in quantum mechanical and classical simulations of nanoscale phenomena and for the bridging of length and timescales for *multiscale modeling and simulation*.
- Computational methods must be devised to identify *quantitative measures for characterizing and monitoring structural order* in nanoscale systems, to *interpret nanoscale characterization measurements* and *monitor nanofabrication and nanomanufacturing* processes.
- Revolutionary improvements in *high-performance computing* and access to fast machines are critical for increasing the accuracy and precision of virtual measurements from *ab initio*, molecular and mesoscale simulation. Concerted improvements in algorithms, software, hardware (processor speed, memory access, etc.), and middleware will all be required in this regard.
- Revolutionary improvements in *high-speed communications* are required to support increased inter-processor communication and data transfer for distributed and parallel high-performance computing; computational steering, interactive visualization, data mining and manipulation, and database management.

THREE-DIMENSIONAL NANOSCALE IMAGING AND SPECTROSCOPY IN HARD AND SOFT MATERIALS

Bennett B. Goldberg, Boston University

The grand challenges in nanoscale measurements in the optical regime fall into several different categories, as discussed below.

Surface Proximity Nanoscale Optical Imaging Status

In the last 5 years, optical nanoscale measurements have vastly improved for several classes of materials and systems. Scanned probe microscopy with tip-enhanced, near-field scanning optical or near-field apertureless techniques are reducing optical resolution down to tens of nanometers for hard materials. Single carbon nanotubes have been resolved spectroscopically with tip-enhanced Raman, and sophisticated optical tips have been used to resolve the distribution of the optical matrix element in single quantum dots. These approaches lend themselves to strongly resonant (to reduce background), sparse systems located within a few nanometers of the surface.

Surface Proximity Nanoscale Optical Imaging and Spectroscopy Challenges

- Approaches for 3D tomography, allowing one to examine subsurface structures to understand how buried systems can be imaged.
- Develop tip-enhanced and aperture and apertureless probes that are reproducible, easily fabricated, and robust. The field of AFM exploded following the introduction of mass-produced silicon and silicon nitride cantilevers. A similar effort toward metal nanorods embedded in tips, solid immersion lenses, and cantilevers needs to be built. This is largely a technological limitation.

Subsurface Far-field Optical Imaging and Spectroscopy Status

In the field of subsurface imaging, where proximity probes can no longer access relevant information, large advances in the past few years have been in development of solid immersion lens techniques. Our group and others have pioneered the use of solid immersion microscopy, and in silicon SILs for subsurface imaging of ICs, we currently have the highest-resolution images in the world in the optical regime. Solid immersion microscopy is being introduced to take silicon processing requirements to the next node and beyond.

Subsurface Far-field Optical Imaging and Spectroscopy Challenges

Development of modeling the process of solid immersion imaging in heterogeneous systems: We know how to calculate optical response in a homogeneous system, but when the lens and substrate material differ, and when the object under interest is close to other heterogeneous materials, we do not yet know how the system will respond.

Development of lenses and lens systems for solid immersion microscopy. The technical challenge is to build SILs and NAILs where the effective surface contact area is small, so that surface roughness is not an issue in imaging. Yet the lens must have very high index, low absorption and broad transmission from the ultraviolet to infrared. These should be placed on easily controlled mechanical systems.

Far-Field Imaging and Spectroscopy in Biological Systems and Soft Materials Status

The last decade has seen the steady growth of techniques and approaches that have continued to improve biological imaging, especially in the area of fluorescent techniques. The advances have fallen into two broad categories; first single molecule imaging, and second, fluorescent techniques for biological systems with sparse, yet large numbers of fluorphores. Advances have seen the development of two-photon techniques by Webb and others, stimulated depletion by Hell and coworkers, and an interesting array of interferometric techniques using patterned excitation, and interferometric excitation and detection. Our group (Goldberg, Unlu, and Swan) has developed spectral self-interference microscopy that has demonstrated 10 nm resolution in one dimension and that we believe will someday provide true 3D biological imaging at 20 nm or so.

Far-field Imaging and Spectroscopy in Biological Systems and Soft Materials Challenges

- *Probes:* Molecular and nanocrystal probes that are brighter and live forever.
- *Detectors:* There is a critical need for hyperspectral arrays. These are CCDs or similar that have spectroscopic capability at each pixel. Having 1,000 points of wavelength information at each pixel would help develop all new interferometric techniques.
- *Modeling:* We need new tools to understand all the influences of a heterogeneous environment on interferometric biological imaging.

DETERMINATION OF SIZE, DISTANCE AND STOICHIOMETRY USING NANOSCALE MEASUREMENT TECHNIQUES

Peixuan Guo, Purdue University

Perspectives in Nanoscale Measurement

Single-molecule FRET (Fluorescence Resonance Energy Transfer) is a cutting-edge technique whereby the distance between two molecules can be determined when they are in close proximity (within 10 nm). One molecule, known as the donor, is excited by a laser. When a second molecule, known as the acceptor, is close enough to the donor, a transfer of fluorescence occurs, and the emission is measured by a specialized apparatus. The efficiency of the energy transferred is proportional to the distance between the donor and receptor. A formula for the calculation of distance has been developed. Based on the amount of energy transferred, the distance between the two molecules can be determined, and other information can be gained, including data on various conformational changes of the particles and the overall dynamics of a given system.

Photoaffinity cross-linking is accomplished through the transformation of chemically inert molecules into reactive states through exposure to ultraviolet radiation. By this method, specific nucleotides, amino acids, chemical groups, or parts of molecules can be labeled and crosslinked to neighboring components within a distance of around 1.2 nm. The identification of cross-linked sites will provide information that this location is away from the photoaffinity-labeled location within 1.2 nm. This method can be used for the measurement of distance both inter- and intramolecularly.

Both electron microscopy and cryo-AFM (atomic force microscopy) involve high magnifications, allowing for the detailed observation of molecules as small as a few nanometers. Such direct observation is useful because it does not involve theoretical extrapolation of the distances between the components in a given system.

Molecular sieves can be used for the measurement of diameter of nanoparticles with defined holes ranging in size from micrometers to nanometers. A simple but valuable procedure using these molecular sieves allows for the determination of whether a particular molecule is larger or smaller than the channel, with given size, of the molecular sieve. If it is larger than the opening in the sieve, then it will be unable to pass through, and if it is smaller than the opening, then it will be able to pass the sieve. Such techniques can be particularly useful when the size of the sieve is close to the size of the molecule in question.

Both binomial distribution and log/log plot have been used to aid in the determination of the stoichiometry of viral and nanobiological assemblages. The concentrations of individual phi29 motor components were varied one by one while holding all other concentrations at optimal levels. The amount of PFU/ml (plaque-forming units) was measured and plotted against concentration on the y-axis. The tangents of the PFU versus concentration curves were plotted, and a best-fit polynomial equation was determined. Subsequent experimental procedures have demonstrated that this equation is an accurate method by which to determine stoichiometry.

JOINT R&D SUPPORTING WORLD STANDARDS: AN INFORMAL REFLECTION

Andy Henson, U.K. National Physical Laboratory

Global Data for Global Standards: Underpinning Single World Standards with Joint R&D (for Measurement and Testing)

As part of a strategic secondment from NPL to NIST the author was asked to consider novel approaches to address the current regional competition in documentary standards, a process that limits market access.

The author hypothesizes that joint R&D on measurement techniques and test methods would greatly assist in achieving the goal of world standards for nanotechnology. A PowerPoint presentation is available. Those wishing to support this initiative are asked to contact the author.

PERCEIVED CURRENT NEEDS AND RECOMMENDATIONS FOR NANOMETROLOGY

Lowell P. Howard, Precera, Inc.

Historical Perspective

Nanometrology as it is currently practiced in national measurement institutes around the world very nearly follows the path laid out by Taniguchi in 1974, when the word nanotechnology was coined [1]. Many *micrometrology* research and working groups eventually underwent a name change in the early 1990s to become *nanometrology* groups. The tools of the trade have all made evolutionary changes so that today PZT actuators (and similar materials), flexure mechanisms, linear motors, and laser interferometers serve in nanopositioning applications, and their commercial availability continues to broaden. In general, selecting a nanocapable sensor or actuator is now an exercise of selecting a product from a catalog. This was certainly not the case 10 years ago, although room always exists for new and improved types of sensors and actuators. So, where are all the breakthroughs, and how do they affect nanometrology, when so often we are using brute-force measurement methods?

Recommendation #1: Research and Qualify More Low-Outgassing Materials

Low outgassing vacuum instrumentation is crucial for reducing the interactions between a specimen and its environment and is important in semiconductor processes, particularly those involving reflective optics. Government can research and place low-outgassing/low-particulate generation materials and packaging information into the public domain. Low-outgassing materials were researched heavily in the 1960s as part of the space program and the best information today exists on the NASA online database [2]. Placing these data into the public domain can speed development and make it easier for small business to contribute to nanotechnology-related fields.

Recommendation #2: Alignment Methods and Self-Calibration Algorithms Should Be Key Research Areas

Develop and place into the public domain multiaxis measurement system alignment methods and techniques that minimize Abbe error, cosine error, and orthogonality errors in all DOFs.

The real issue for nanometrology (length-based) is not interferometer resolution, sensor resolution, or even index of refraction measurement and compensation, it is alignment. Nothing kills an uncertainty budget faster than an Abbe error. Until one actually goes through the procedure of determining an uncertainty budget for a real-world nanomeasument, this fact is easy to overlook. Continued education efforts on the topic of uncertainty budgets can help to educate the next generation of nanotechnologists.

Proprietary knowledge and methods exist in the private sector for aligning inspection machines, ebeam writers, and wafer steppers using self-calibration algorithms. Placing such algorithms into the hands of

nanotechnologists via publicly funded research can help prevent others from reinventing existing methods as they develop the next generation of nanomanufacturing tools.

Recommendation #3: Traditional Mechanical Measurements Are Not Getting Any Easier; Make Sure That Basic Measurements Are Done Right

Continue to fund programs to provide SI-traceable pitch, line width, and length-scale measurements. Given the huge development costs for a new technology, physical standards from a National Measurement Laboratory frequently lag behind the current state of the art for lithography, but they are essential to provide a bedrock of support for test and measurement instruments.

Recommendation #4: Low Force Calibrations and Standards Are Needed

Continue to fund research for improving the accuracy and resolution of force measurements. NIST has an existing program in this field, and the agency should continue it. The program will be essential to the future of an accurate nanonewton-class measurement that will be of importance to probe microscopy.

Summary

Bottom-up nanotechnology might happen in a fluid well on a glass plate, but measurements are still made in a top-down fashion using large instruments. In fact, the design of an instrument capable of measuring to nanometer-level uncertainties on an object sized anywhere larger than 100 mm is just about as difficult as designing a small spacecraft. Structural dynamics, thermal and power management, and the use of exotic materials to raise structural resonant frequencies are all needed design disciplines. Given that aerospace projects are undertaken by large groups, perhaps nanotech projects will experience a similar upsizing as needs progress out of the laboratory.

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1. N. Taniguchi, On the basic concept of nano-technology, *Proc. Intl. Conf. Prod. Eng.* Tokyo, Part II, Japan Society of Precision Engineering, 1974. Author's note: I have to like Norio Taniguchi's definition of nanotechnology. He was a production expert and he talked about the atom as the smallest practical unit of stock removal (yes, stock removal).
2. The NASA outgassing site is at http://outgassing.nasa.gov and contains a searchable database of low-outgassing materials, some (but certainly not all) of which are useful for vacuum work.

SCANNING PROBE-BASED ELECTRICAL MEASUREMENTS

Julia W. P. Hsu, Sandia National Laboratories

Scanning probe microscopy (SPM)-based techniques are natural choices for local characterization at the nanoscale. In particular, it is often useful to probe electrical properties in nanoelectronic devices. The major advantage of using SPM is that local electrical properties can be directly correlated with structural properties through simultaneous topographic measurements and possibly with other physical properties. Scanning tunneling microscopy (STM) and conducting-probe atomic force microscopy (CP-AFM) are two common methods. STM has the advantage of true atomic resolution. However, the sample needs to be conducting and the imaging environment is usually in ultra-high vacuum. These requirements limit the usage of STM to mostly research problems.

CP-AFM is much more "user friendly" in comparison. It uses a standard AFM tip that is coated with a desired conducting film, although some specialty tips use a solid conductor rather than a coating. Depending on the bias and imaging conditions, different electrical properties can be measured. The most straightforward example is to perform two-point current-voltage (I-V) measurements locally, in which the conducting tip is one electrode [1]. An extension of local I-V measurements is to use the tip as a gate to control current flow; this is particularly useful for carbon nanotubes [2]. The CPAFM can also be used to measure local capacitance and impedance [3] in general, and surface potential variations near defects [4] and along source

and drain in nanotransistors [5], dopant distribution in devices [6], and trapped charges down to the single-electron level [7].

Over the last decade, standard AFM has moved out of research laboratories and has become a standard characterization tool in fabrication facilities and on manufacturing floors. Many of the electrical "modes" discussed above are now available in commercial instruments. However, some challenges remain to be met for SPM techniques to have a true effect on nanoelectronics.

- *Speed and size:* Current technology limits us to ~10 Hz/line and ~100 μm in size. Ideally, AFM should be more like scanning electron microscopy with the zoom in/out dynamic range and ease at video scan rate.
- *Sensitivity and resolution:* Because the lowest detectable signal is limited by noises, which are independent of probe size, the SPM-based current measurements have lower sensitivity in current density and capacitance measurements are more prone to stray capacitance. Resolution is often a trade-off with sensitivity. A smaller tip produces a smaller signal, which is more difficult to detect. Also, a sharper tip is more susceptible to wear and damage.
- *Quantitative results:* This must be accomplished through realistic modeling of experimental results. In SPM, the role of the tip is often not passive [8], and the interaction between the tip and sample need to be considered.
- *Interface vs. surface:* Ballistic electron emission microscopy is an STM-based technique that can uniquely probe the electrical properties of buried interfaces, but its applications are limited to a few fairly ideal systems.

Correlation between chemical (compositional) and physical properties: Typical techniques for chemical characterization (e.g., Auger and SIMS) have sensitivity that can detect impurity concentrations greater than 10^{-16} cm^{-3} under the best conditions, and typically 10^{-18} cm^{-3} with micrometer-size beam size. However, electrical properties change drastically in this range. A more accurate measurement of chemical properties is desired. Chemical-sensitive AFM with molecular-layer coated tips is still in its infancy.

Because surface (interface)-to-volume ratio is much higher in nanomaterials, the control and understanding of surfaces (interfaces) are absolutely critical. One example is electrical contact. In conventional semiconductors, "ohmic" contacts are made according to recipes formed through trials and tests, typically involving specific surface chemical cleaning procedures, vapor deposition of metal(s), followed by thermal annealing. These contacts often have a transition region that contains alloys of the semiconductor and the contact metals. This type of approach is not applicable for nanoelectronics because the active regions are too small and chemically and mechanically too fragile for such treatments. Thus, there are urgent needs for new methods to form electrical contacts, new characterization tools to examine them, and new body of knowledge.

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SUMMARY OF THE GRAND CHALLENGE WORKSHOP ON NANOMATERIALS

Robert Hull, University of Virginia

At the NSF-sponsored workshop on "Grand Challenges in Nanomaterials" in Arlington, Virginia in June 2003, about 70 research leaders in the field met to discuss, explore, and identify future new directions and critical needs for the next decade and beyond. The key pervasive theme that was identified was the need to develop techniques for assembly of nanoscaled materials over multiple length scales, at the levels of efficiency, economy, and precision necessary to realize broad new classes of applications. This could enable major advances in such diverse technologies as electronics, computation, telecommunications, data storage, energy storage/transmission/generation, health care, transportation, civil infrastructure, military applications, national security, and the environment. Elements of this strategy include development of new self-assembly and lithographic techniques; biologically mediated synthesis; three-dimensional atomic-scale measurement of structure, properties and chemistry; harnessing of the subatomic properties of materials such as electron spin and quantum interactions; new computational methods that span all relevant length- and timescales; a fundamental understanding of acceptable/achievable "fault tolerance" at the nanoscale; and methods for real-time and distributed sensing of nanoscale assembly. A parallel theme was the need to provide education concerning the potential, applications, and benefits of nanomaterials to all components of society and all levels of the educational spectrum. This presentation summarizes the conclusions and recommendations from this workshop.

INSTRUMENTATION AND METROLOGY FOR NANOTECHNOLOGY: THE RELATION TO ENVIRONMENTAL AND HUMAN HEALTH PROTECTION

Barbara Karn, U.S. Environmental Protection Agency

Metrology supports industry by enabling the benefits of new products and processes to be measured and by stimulating new product development in the instrument sector in addition to raising productivity through improved process and quality control. Measurement also provides a foundation for environmental and human health protection. Four important topics comprise metrology and the uses to which its results are applied — written standards, scientific instrumentation, validated measurement procedures, and measurement standards.

Protecting the environment relies on written standards. Limits are placed on emission of pollutants; air, to be healthy, must meet certain standards; both drinking water and surface waters have quality criteria; pollutants are measured and controlled from landfills. Metrology is necessary to enable the measurements that form the basis for deciding environmental standards. Currently, there are no standards for nanosized materials in the environment. Particularly in the air, nanoscale particulate matter may have an effect on human health. However, the inability to measure ambient nanoparticulates quickly, inexpensively, and accurately severely restrains research that would lead to intelligent standards for airborne nanoparticles that might affect human health. Waterborne nanoparticles may likewise need standards if they are found to be harmful to aquatic organisms.

Scientific instrumentation is essential in examining nanoscale materials and their interactions and impact on and in the environment. In addition, nanotechnology itself can form the basis for detection of other materials. New instruments using massively parallel nanoscale sensor arrays could enable more sensitive, highly selective detection of environmentally important analytes including both chemical compounds and biological organisms such as algae, bacteria, or viruses. Instrumentation is needed for monitoring nanomaterials to determine their fate, transport, and transformations.

Validated measurement procedures are necessary for quality control in monitoring and analyzing environmental samples. If emissions standards are necessary, there must be reasonable means for affected industries and organizations to measure nanoparticles for compliance requirements. Measurement standards are essential for research to proceed in nanotechnology related to the environment. If there is no standardization, there is no comparability among the numerous research laboratories involved in examining nanoscale science and its relation to the environment. Measurement standards are also needed for quality control during monitoring.

The importance of measurement in the environment at the nanoscale was discussed at the NNI workshop on the Nanotechnology Grand Challenge in the Environment, May 8–9, 2003. One of the five major discussion topics was nanotechnology applications for measurement in the environment as applied to sensors, monitors, models, separations, detection, fate and transport, data gathering, and dissemination. The vision statement of this workshop group says: "The unique properties of nanoscale materials will enable the development of a new generation of environmental sensing systems. In addition, measurement science and technology will enable the development of a comprehensive understanding of the interaction and fate of natural and anthropogenic nanoscale and nanostructured materials in the environment."

Research needs were identified in five areas: (1) biological sensor technologies that are sufficiently stable to allow detection *in situ* on a continuous basis for high-density usage; (2) a general "array" for detection of a wide variety of potential analytes; (3) information concerning the diversity of chemical composition at the nanoparticles level, and the transformations that occur and measurement techniques that distinguish the chemical composition of particle surface layers from the particle interior; (4) generic nanoscale assembly methods; and (5) advances in spectroscopic instrument technologies that allow rapid detection of low signal strength, while probing smaller volumes of a nanoparticulate sample.

Measurement is fundamental to the progress and quality of all scientific endeavors and engineering applications. It provides an underlying foundation for research in environmental and human health protection. Advances in metrology to both measure nanoscale materials and to use nanotechnology in measurement go hand in hand with advancing the protection of the environment and human health.

SCALE-BRIDGING METROLOGY FOR NANOMECHANICS

Kyung-Suk Kim, Brown University

Metrology for nanomechanics is critically needed to set various standards in emerging nanotechnology as well as to advance research in nanoscale science and engineering. Because metrology is to measure variations of positions of matters in time and space, and forces associated with such motions, appropriate control and resolution have to be established in measuring such motions of nanometer-scale objects. In particular, such control and resolution are needed in measuring multiscale phenomena in nanomechanics. Therefore, scale-bridging metrology has to be developed for nanomechanics. Examples of such metrology will be discussed, including scanning probe microscope (SPM) interferometry for measuring deformation of nanoscale objects, the Field Projection Method for measuring deformation, stress and state of energy partitioning in nanoscale objects, high-sensitivity and low-noise curvature measurements for evaluating nanoscale stress distribution, (AFM/--/SFA) scale-bridging force measurements for identifying mesoscale tribological mechanisms, high-resolution EBSP for measuring texture evolution of nanocrystalline materials, high-resolution transmission electron microscopy coupled with the field projection method for measuring deformation of defect core structures, and noninvasive force measurements for biological molecular and cellular motions. In addition, high temporal and compositional resolution measurement of nanostructural evolution will be discussed as well.

Examples of scale-bridging metrology that has to be developed include:

- Scanning probe microscope (SPM) interferometry for measuring deformation
- Field projection method for measuring deformation/stress and energy partitioning
- High-resolution transmission electron microscopy coupled with field projection for measuring deformation of defect core structures

- High-sensitivity curvature measurement for evaluating nanoscale stress distribution
- (AFM/--/SFA) scale-bridging force measurements for identifying mesoscale tribological mechanisms
- Noninvasive force measurements for biological molecular and cellular motions
- High temporal/compositional resolution measurement of nanostructural evolution, including surface roughness evolution spectroscopy
- High-resolution EBSP for measuring texture evolution of nanocrystalline materials

IMPORTANCE OF METROLOGY TOOLS FOR NANO CHARACTERIZATION

Leslie Kramer, Lockheed Martin

Although my background during undergraduate and graduate education as well as my initial professional responsibilities primarily dealt with ferrous metallurgy, my research interests over the past decade have targeted the engineering applications, structural characterization, and metrology of carbonaceous materials such as micrometer- and sub-micrometer-sized polycrystalline diamond, carbon fiber reinforcements for resin composites, and single-wall carbon nanotube ropes. Ever since I received a sample of nanotube buckypaper from Dr. Smalley in 1997, my research effort has continually focused on resin infiltrating nanotube buckypapers for the purpose of exploiting small quantities of this reinforcement to produce reasonably sized panels of nanocomposites in engineered structures such as missile wing airfoils. Thanks to AFRL sponsorship over the past 3 years, Lockheed Martin in conjunction with the Florida A&M University-Florida State University (FAMU-FSU) College of Engineering has succeeded in designing and fabricating aerostructures made from a few layers of resin infiltrated buckypaper instead of conventional carbon fiber composites. To keep both the cost and structural weight low, only wing skins of appropriately 50 micrometers in thickness are generally fabricated, although both random and magnetically aligned nanotube configurations have been demonstrated. Buckypapers have been produced over 400 mm in length and 100 mm in width.

Lockheed Martin and our academic partner (FAMU) could not have accomplished the consistent manufacture of nanotube-based aerostructures without accurate metrology tools for nanocharacterization. Because of these tools such as atomic force microscopy and focused ion beam, we could not have conducted a “design of experiments” to produce consistent open-cell structures in three dimensions with reasonable rope diameter statistics that allow resin infiltration during resin transfer molding. These experiments have been validated to a degree by molecular dynamic simulation software. Over the next 10 years, we expect metrology tools to improve to allow single nanotube placement, the insertion of nanoelectronics into nanostructures, and the ability to separate and size bundles, clusters, and individual particles on the nanoscale. In addition, the ability to obtain physical and mechanical measurements on these material forms will be mandatory to the custom material design practices.

FROM MICRO- TO NANOSENSORS: DO WE NEED PARADIGM CHANGES?

Mihai N. Mihaila and Cornel Cobianu, Honeywell Romania, Cleopatra Cabuz, Honeywell International

The classical way to improve the performances of solid-state microsensors is to resort to the refinements of the microtechnology specific methods. However, increased performance requires, in many situations, size reduction. In a first instance, this could be a source of large variation from a sensor to another. Moreover, this trend can be a source of erratic behavior for the same physical system. However, by decreasing the size one can reach thresholds at which the surface to volume ratio becomes so high that the performances of the system are almost entirely determined by its surface properties. Therefore, the surface would become of paramount importance in dictating the state of the art. Consequently, in spite of the formidable research in the surface science, one can ask whether the knowledge accumulated so far would be enough to offer answers to the future challenges.

Although there are still resources to be exploited in microtechnology, the demand for increasing performance already involves nanotechnology specific approaches. This is one of the main goals in the next 10 years in

improving sensor performance. In this respect, one can improve the classical structure by using nanomaterials, such as, for instance, nanoparticle films or carbon nanotubes, as in the case of some gas sensing. Nanomaterial-surface interaction and distribution of stress in nanomaterial or nanomaterial specific area are other factors of concern in the future sensing systems. For instance, interaction between carbon nanotube, nanoparticles or quantum dots with the substrate they are sitting on (e.g., silicon dioxide) involves the substrate into the conduction phenomena in a possibly unpredictable way. This interaction is also a source of noise, an important limiting factor in microsensors. It can become even more important in nanosensors, where its intensity could vary from sample to sample, therefore affecting the reproducibility. In this respect, examples will be given of how nanoparticle films of almost identical conductivity feature noise levels differing by orders of magnitude. This situation could be critical for nanosensors with a few nanoparticles in the active area. On the other side, putting the nuisance to work, noise can be used to increase the sensing properties, as in the case of stochastic resonance-based nanosensor [1]. It illustrates the idea that searching for new sensing principles is an important challenge in the future. In this respect, many important contributions are expected from molecular dynamic simulations [2].

Because macroscopic averaging could be a source of information loss, another challenge would be to identify the macroscopic "messengers" with the richest microscopic information. For instance, in the case of some nanoparticle films, conduction measurements point to the validity of the Ohm law, therefore, the system behaves linearly, while noise measurement indicates that the nature of the conduction is strongly nonlinear [3]. Related to this is the question whether the macroscopic physical laws are still valid at nanoscale.

Because of its infinitely small active area, the response of a nanosensor can be dominated by a single structural defect. In this case, one expects that the signal from solid-state nanosensor to be dominated by so-called blinking or random telegraph noise. Extracting information from this signal could be unreliable because, in this case, the time average is no more equivalent with the ensemble average (violation of the ergodic hypothesis). Also, if processed in different ways, the information content of the same signal we get from the nanoworld could be different. Consequently, changes are expected in our current methods of measurement and characterization, even if they are noninvasive. These statements and questions indicate that the transition from micro- to nanosensor is not always smooth. Consequently, important paradigm changes are expected.

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ATOMIC-SCALE ELECTRON MICROSCOPY AND SPECTROSCOPY OF INDIVIDUAL DOPANTS, DEFECTS AND INTERFACES IN NANOMATERIALS AND DEVICES

David A. Muller, Cornell University

Atomic-resolution electron microscopy and spectroscopy can now determine composition and electronic structure not only of individual nanostructures but also of individual defects, interfaces, or atomic columns inside those nanostructures [1, 2]. The sensitivity and resolution extends to the imaging of single dopant atoms or vacancies buried inside their natural environments, allowing us to study the early stages of precipitate nucleation and identify the clusters responsible for electrical deactivation in integrated circuits [1, 3]. In fact, the smallest feature in a modern transistor, the gate oxide, is already little more than an interfacial layer just over 5 atoms thick, and the fundamental limits to device scaling are set by the measured electronic structure—determined by atomic-scale electron spectroscopy [4].

Modern developments in electron microscopy and spectroscopy have placed within reach the ultimate goal of knowing the location, chemical, optical, and electronic properties of every atom in a nanostructure. Since the early 1990s, it has been possible to map chemical and bonding information at the atomic scale, using a scanning transmission electron microscope (STEM) [5-7]. Electron energy loss spectroscopy (EELS) reveals detailed information about the local electronic structure, and in particular the conduction band density of states partitioned by site, angular momentum, and atomic species. With improvements in detector efficiency it has become possible to apply this spectroscopy to the study of grain boundaries and buried interfaces using 2–5 Å wide probes [8, 9].

More recently, we have obtained the first images of single impurity atoms inside a crystal [3], a task requiring extraordinarily smooth samples and a quiet measuring environment. These are key enablers for work on dopant deactivation, device scaling, and the chemistry of oxide heterostructures [1-4]. Combining these methods with a high-energy-resolution spectrometer on a monochromated electron microscope column and aberration correctors [10] enables us to perform subnanometer (and possibly subangstrom) energy loss measurements on a scale relevant to optical and electronic properties.

Breakthroughs in instrumentation and algorithms have dramatically changed the field of electron microscopy, opening new areas from the imaging and spectroscopy of individual dopant atoms and clusters to 3D tomography of nanoparticles, viruses, and biological structures. Early results in subangstrom resolution and millivolt spectroscopy are now being applied to nanoscience problems, and the national initiatives in aberration-corrected instruments (such as the TEAM project) should make such facilities widely available. Areas where electron microscopy provides unique tools include:

- Three-dimensional electron tomography for nanoparticles, semiconductor devices and biological materials, often at subnanometer resolution
- Electron holography for mapping nanometer-scale magnetic and electric fields in structures
- Imaging of single atoms, clusters and vacancies buried inside materials
- Electron spectroscopy at high spatial and energy resolution for subnanometer chemical and optical properties
- Smaller than an atom: aberration-corrected microscopes for imaging with subangstrom resolution.
- Imaging individual molecules and defects in biomaterials
- *In situ* microscopy using environmental cells of deformation and growth (even in liquids)

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METROLOGY NEEDS FOR MAGNETIC NANOPARTICLES: HOW TO MEASURE THE MAGNETIC PROPERTIES OF INDIVIDUAL MAGNETIC NANOPARTICLES?

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The science of magnetic nanoparticles (3–10 nm) has been growing rapidly because of magnetic recording and biomedical applications. The particles present two different metrology problems (structural and magnetic characterization). The particle size range pushes the limits of electron microscopy for structural characterization. Local electrode atom probe could provide 3D atomic positional information with near-atomic resolution.

$L1_0$ FePt nanoparticles are a serious candidate for future magnetic recording media with data storage densities beyond 1 terabit/in^2. We pursue basic research to solve the materials science problems that would allow these particles in be used in future disk drive with terabyte data storage capacities. We have prepared FePt, CoPt, FePd, FeCoPt, FeCuPt, FePtAg, FePtAu and FePdPt nanoparticles. The particles have an fcc structure and are superparamagnetic with a narrow distribution of particle sizes. Casting dispersions onto silicon wafers gives self-assembled films consisting of close-packed arrays. The films must be heated to temperatures above 550°C to transform the particles to the $L1_0$ phase, having high magnetocrystalline anisotropy, giving ferromagnetic films. We have shown that FePtAg or FePtAu nanoparticles can be transformed by heating at lower temperatures. During heating the Ag or Au leave the particles, creating lattice vacancies, and thereby allowing the Fe and Pt atoms to move to their $L1_0$ lattice positions.

High-resolution EDX analysis on films containing as prepared FePt nanoparticles revealed a distribution of particle compositions. Although the chemistry to prepare FePt nanoparticles gives narrow particle size distributions, the composition varied from particle to particle. A distribution of particle compositions was also observed for FePtAu nanoparticles. Measuring the composition of individual particles having diameters of 3–4 nm is very difficult, beyond our capability, and we collaborate with some of the best electron microscopy groups in the world (Hitachi Maxell and J. Chapman at U. of Glasgow). It would be useful to find routine methods to determine the composition of individual nanoparticles.

The ultimate of high-density limit magnetic recording technology is expected to be recording in monolayer films of FePt nanoparticles, where one bit is recorded in a single particle. The most fundamental problems are how to record and read at this extreme spatial resolution. Furthermore, we have a distribution of particle compositions, and that sintering occurring during heat treatment gives rise to a distribution of particle volumes. Magnetic characterization tools currently available (VSM and AGM) measure the magnetic properties of a large collection of particles. Our ability to extract information from these measurements is limited by the need to account for the unknown distribution of magnetic properties. The metrology challenge is how to measure the magnetic properties of individual particles (e.g., 3.5 nm diameter $L1_0$ FePt nanoparticle with volume of 2.2×10^{-20} cm^3).

Magnetic nanoparticles have potential biomedical applications including detection of biomolecules, separation of biomolecules and cells, MRI contrast enhancement, and hyperthermia therapy. For example the Naval Research Laboratory has developed the Bead Array Counter (BARC biosensor) for the rapid, multianalyte detection of biowarfare agents. Beyond detection of biomolecules is the use of magnetic fields to manipulate biological processes. To develop this science it would be useful to manipulate individual nanoparticles to place them precisely in a desired location.

NEAR-FIELD OPTICAL METROLOGY AND NANOCHARACTERIZATION

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Progress in science and technology was often triggered by the invention of new instrumentation. Because of the availability of new kinds of microscopes and spectroscopic techniques we have developed a thorough understanding of physical phenomena, ranging from the atomic structure to the structure of biological cells. In the words of Nobel Laureate Rosalyn Yalow "*New Truths become evident when new tools become available.*" Also, the rapid advance of nanoscience is largely a result of our new instrumentation that allows us to manipulate and measure structures on the nanometer scale.

In nanoscience one often first attempts to understand the nanoscale building blocks in isolated form before assembling them into a functional device (bottom-up approach). However, the properties of the building blocks can change once they are embedded into a macroscopic structure. This change is caused by interactions between the building blocks and also interactions with the environment. In fact, one of the most interesting aspects of nanoscale systems involves properties dominated by collective phenomena. In some cases, collective phenomena can bring about a large response to a small stimulus. To study nanoscale systems in a complex environment it is necessary to develop spectroscopic techniques with high spatial resolution.

Important instruments for the characterization of nanoscale materials are electron microscopy and scanning probe microscopy. However, without any prior knowledge about the specimen it is often difficult and challenging to identify the constituent parts of the specimen and thus to learn about its functionality and the underlying physics. This is mainly because electron microscopy and most scanning probe techniques render high-resolution topographical images with poor molecular (chemical) specificity.

In contrast, optical spectroscopy provides a wealth of information on structural and dynamical properties of materials as the energy of light quanta (photons) are in the energy range of electronic and vibrational transitions in matter. Combining optical spectroscopy with microscopy is especially desirable because the spectral features can be spatially resolved. Unfortunately, the diffraction limit has prevented researchers from resolving features smaller than half a wavelength of the applied radiation. However, in recent years a novel technique, called near-field optical microscopy, has extended the range of optical measurements beyond the diffraction limit and stimulated interest in many disciplines. With near-field optical microscopy, resolutions of 100 nm are nowadays routinely achieved. Pushing the resolution of optical microscopy down to 10 nm would benefit both biological and materials science because an instrument with 10 nm resolution would allow direct imaging and characterization of individual biological proteins and would enable us to image quantum wave functions (orbitals) in semiconductor nanostructures. Optical radiation can penetrate through matter and is therefore well-suited for subsurface characterization.

NANOSCALE CHARACTERIZATION BY SCANNING PROBE MICROSCOPY: CHALLENGES AND OPPORTUNITIES

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A number of techniques can potentially provide capabilities for nanocharacterization, yet only two are in widespread use—electron microscopy (EM) and scanning probe microscopy (SPM). SPM is the leading candidate from cost and sample preparation perspective. SPM provides unique characterization capabilities with true nanometer-scale resolution on a variety of samples. The types of surfaces that SPM typically investigates range from semiconductor nanostructures, to soft polymers, to biological tissues and cells. The most important challenge faced by SPM techniques is that they typically provide no real surface composition information. Other challenges include variability in probe size, shape, and lifetime; inability to image high-aspect ratio, fragile, or very soft structures; and low throughput determined by the limitations of the scanning speed and range.

Technological Approaches to Overcoming Challenges in SPM

Probe limitations: One of the most important technologies for boosting SPM capabilities is carbon nanotube AFM. Use of carbon nanotubes as AFM probes promises to reduce the effective size of the tip as well as unwanted adhesive interactions and boosts the aspect ratio of the tip. Challenges to be overcome include refining the fabrication process to produce uniform and stable probes and adapting the process to mass-production.

Throughput: SPM is inherently a serial technique that acquires images point by point; therefore, the throughput gains may come from increasing scanning speed or parallelization of imaging process. Speed improvements typically come from using shorter cantilevers with higher-resonance frequencies [1] or from other improvements in the AFM bandwidth [2].

Chemical composition: One approach is based on controlling and monitoring probe-sample interactions. It has been demonstrated that chemically modified AFM probes can reliably distinguish between hydrophobic and hydrophilic samples, monitor local pH changes, and provide quantitative estimates of local surface energies [3]. Chemical modification can also be combined with carbon nanotube AFM tip technology to produce the "ultimate AFM probe" [4]. Researchers have developed approaches that use energy dissipation resulting from probe–sample interactions to map surface composition [5, 6]. The leading challenge for CFM is developing more robust chemical coatings and modifications that will enhance chemical discrimination.

Near-field scanning optical microscopy (NSOM) is another candidate approach [7] but suffers from using a single probe to collect topographical and optical information. As the result, high spatial resolution hurts optical throughput. Another approach combines AFM with optical techniques such as confocal microscopy [8]. However, this approach may not resolve optical signatures of the surface features that are located within the diffraction limit. Another promising approach combines APM with surface-enhanced Raman spectroscopy (SERS). Researchers have proposed placing Raman-enhancing nanoparticles on the SPM probe [9] and use of local field enhancement by SPM to improve resolution of fluorescence imaging [10].

Nanoscale interaction force measurements: Characterization of the interactions between nanostructures is as important as characterization of surface morphology and chemical composition. SPM has already been successful in measuring interaction forces on truly microscopic scale. A short-term problem is posed by the lack of robust and accurate methods for calibration of the force measurements: standard calibration techniques produce about 10% error. The first step for standardization could be the establishment of a common molecular scale force standard for probe. In the longer term, we still need to push the force resolution of the cantilever systems down into the single piconewton regime, all while maintaining adequate cantilever stiffness to avoid jumps. Drastic reductions in the instrument noise level and use of shorter cantilevers should drive progress in that area. We will also need to expand the AFM to probe different loading rates and regimes. Interaction force measurements often require large statistics; therefore the throughput issues are very important. A long-term challenge is using SPM for direct mapping of full-energy landscapes (perhaps based on using thermal-noise assisted probing). Such capability should then open up a way for a truly rational design of nanoscale assemblies.

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SUBANGSTROM ELECTRON MICROSCOPY FOR SUBANGSTROM NANOMETROLOGY

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The revolution in nanoscale science and technology requires instrumentation for observation and metrology—we must be able to see and measure what we build. Because nanodevices operate on the level of a few molecules, or even a few atoms, accurate atomic-scale imaging is called for. *High-resolution aberration-corrected electron microscopes (both TEM and STEM) can provide valuable measurements at the subangstrom level.*

In general, resolution is accepted as the ability to determine whether an image feature represents two objects rather than one. Rayleigh's original resolution criterion, the accepted standard in optics, was derived as a yardstick for judging when two sources of light (stars) could be distinguished from a single source [1]. In the field of microscopy, resolution has continued to be based on the ability to determine if detail in an image represents distinct (separated) objects. In high-resolution electron microscopy these objects are atoms. A resolution of |d| is achieved when atoms separated by a (projected) distance |d| can be perceived as separate objects. Although demonstration of resolution |d| requires the presence of the corresponding spatial frequency 1/|d| in the TEM image spectrum, the mere presence of the 1/|d| frequency is not sufficient to validate a corresponding resolution of |d| [2]. Similarly, in a scanning transmission electron microscope (STEM), a probe size of |d| is a necessary, but not sufficient, requirement to achieve a resolution of |d|.

Test samples with atoms separated by known amounts can be used to measure subangstrom resolutions. Specimens with diamond cubic and zincblende structures can be oriented to provide pairs of atoms in "dumbbell" configurations with well-characterized separations ranging from 1.6 to 0.5 Å. To properly characterize nanomaterials, it is important to be able to see all the atoms, even the light ones. Improved resolution has the advantage of narrowing the peaks corresponding to heavy atoms, thus allowing the lighter atom peaks to become visible. The One-Angstrom Microscope (OAM) at the Department of Energy's National Center for Electron Microscopy (NCEM) at Lawrence Berkeley National Laboratory has demonstrated that subangstrom resolution gives us the ability to image lithium, the lightest of all metal atoms. Characterization of catalyst nanoparticles requires knowledge of the internal structures of the particles as well as their shapes. As well as revealing atom column positions, subangstrom phase-image microscopy can also provide a good estimate of the number of atoms making up each column in a single-atom-species particle [3].

Over the next decade, extension of TEM and STEM resolutions to half-angstrom levels by next-generation aberration-corrected electron microscopes will advance the capabilities of these essential tools for atomic-scale structural characterization [4]. Because improvements in resolution allow for separation of atom columns in many more projection directions, these microscopes will provide much improved 3D characterization of the shape and internal structure of nanodevices and catalyst nanoparticles (perhaps even true 3D imaging), and hence provide essential feedback in the nanotheory/construction/measurement loop [5].

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CHALLENGES FOR NANOMECHANICS OF BIOLOGICAL SYSTEMS

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Biological systems are one of the most difficult classes of materials to study mechanically at the nanoscale [1-3]. Their complex multilevel and multicomponent structures (e.g., tissues, cells, proteins) need to be highly purified and characterized with minimal sample preparation damage (if possible, with no polishing and chemical treatments) to give information relevant to *in vivo* function. Nanomechanical testing can be challenging because of the need for near-physiological conditions (i.e., aqueous salt solutions, ionic strength = 0.15 M, pH = 7.4), the existence of varied 3D geometries and multiple buried interfaces, and their dynamic and sometimes, extremely soft, fluid-like nature. New paths in this field are described below.

Integration of nanomechanical testing methods with nanoscale chemical/structural characterization techniques down to the single-molecule level in near-physiological conditions: One promising example of this is the combination of atomic force microscopy (AFM) and surface enhanced Raman spectroscopy (SERS) [4-8]. SERS is a higher-resolution version of traditional Raman spectroscopy, in which the wavelength and intensity of inelastically scattered light from molecules is measured. These wavelengths are shifted from the incident light by the energies of molecular vibrations and hence allow for chemical identification and characterization of substances. Because AFMSERS should be able function in aqueous media, it will provide information about biomolecular identity, bonding, dynamics such as real-time molecular conformational changes (e.g., mechanically induced single protein unfolding), orientation, the spatial distribution of proteins in living cell membranes, the quantification of normal and abnormal proteins in tissue, and chemical variations across nanoindentation sites in whole tissues.

Integration of nanomechanics technologies with high-throughput biological arrays: High-throughput biological arrays such as DNA [7], catalytic RNA [9], protein [10], and live cell arrays [11] have been developed in the context of rapid DNA sequence analysis, platforms for pharmaceutical drug development, fundamental tools to study cell fate and function, and so on. Such arrays have not yet been fully exploited in the context of nanomechanics. For example, high-throughput testing of live, isolated, individual chondrocyte cells [12] can provide information on the self-assembling and nanomechanical properties of the precartilage tissue layer called the *pericellular matrix,* which coats the chondrocyte. Selective deposition of different types of molecules and cells with higher spatial resolution and high-throughput nanomechanical experimental automation, data acquisition, analysis, and archiving are necessary. Once again, combining with nanoscale biochemical/structural assays will provide even further information.

High-resolution chemical characterization of bioactive, chemically functionalized nanosized probe tips: Probe tips functionalized with proteins, ligands and receptors, cells, and nanotubes have enabled studies of biologically relevant intermolecular interactions [13-16]. Probe tip functionalization has been achieved by covalent immobilization, nonspecific physisorption, and conventional adhesives for larger structures (= 1

μm) [17-21]. However, attachment of smaller structures, such as macromolecules, to the apex of a probe tip with a prespecified orientation, conformation, and density is difficult because of the small surface area involved and, for polyelectrolytes, the presence of fixed charge groups. Functionalization and subsequent characterization of parameters such as the polymer chain grafting density, in the vicinity of the probe tip apex, are also difficult and critical to the interpretation of nanomechanical data relating interaction. One effort in this direction have been the use of fluorescence microscopy directly on functionalized cantilever probe tips to detect the presence of bound biomolecules [22]. Another more quantitative method recently developed [23] is the use of chemical force microscopy for this specific application. For example, the nanomechanical interaction between a biochemically functionalized probe tip and a surface of known nanomechanical properties is fit to a standard theoretical model where one or two of the fitting parameters are obtained that represent the density or orientation of the molecules on the probe tip [23].

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NANOMETROLOGY CHALLENGES FOR SINGLE-WALLED CARBON NANOTUBES IN SOLID AND LIQUID PHASES

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After almost a decade of research, the production of Single-Walled NanoTubes (SWNTs) is now being scaled up and "large" quantities of material are being produced (e.g., through the HiPco reaction [1, 2]). This increased availability of SWNTs has enabled the first steps toward making macroscopic materials composed primarily or solely of SWNTs. SWNTs have now been processed into fibers and sheets by solution spinning (or casting) starting from surfactant-stabilized dispersions [3-6] or liquid-crystalline mesophases of SWNTs in acids [7-9]. These two steps—production and processing—are opening routes towards commercialization of SWNT-based materials. To rationalize, optimize, and scale-up production and processing, two metrology problems must be addressed: (1) what are we producing? (2) what are we processing?

The first problem has been studied for several years by a number of research groups, and characterization methods have been developed that are based on SEM and TEM for morphological information, TGA for the quantification of impurities [10] (possibly in combination with purification methods), and combined optical and Raman spectroscopy [11]. The problem is determining SWNT length and length distribution. AFM has been the method of choice and has yielded some length information [12]; drawbacks are extreme sensitivity to sample preparation, as SWNTs of different lengths may stick differently to the substrate, SWNTs may form small bundles during sample drying, and collecting statistically large samples is time consuming. We have developed an alternative length measurement based on intrinsic viscosity measurements of very dilute suspensions of individual SWNTs in acids [8] or in surfactants and water. We are currently extending this method to extract information on the SWNT length distribution.

The second problem is in early stages because surfactant-based processing appeared 3 years ago, and the acid-processing route is even more recent. To understand processing, we need compositional information on the starting material and on the phase behavior and rheological (flow) regimes of the SWNTs in liquids. Such information is needed over a large range of length scales, as SWNTs have been shown to self-assemble into liquid-crystalline mesophases when dissolved at high concentration in sulfuric and chlorosulfonic acids [8]; such mesophases may be present also in the surfactant-stabilized systems. Mechanical rheometry will be key for characterizing the behavior of these SWNTs suspensions; however, it should be accompanied by (nano, micro, and meso) structural studies—that is, scattering and possibly optical (light) and cryo-electron microscopy.

Scattering studies are proving particularly challenging: light scattering may be useful for determining the length of the SWNTs at very dilute concentrations, but it is unlikely to work at higher concentrations. X-ray scattering (XS) will have limited applicability in the sulfur-containing acid systems because of excessive attenuation but should be applicable to the surfactant-based systems. Neutron scattering (NS) is viable for both surfactant-based and acid-based systems, although data collection at low concentration (few hundred ppm SWNTs) is painstakingly slow. The high polydispersity in diameter and length of the SWNT samples presents particular challenges to the *analysis* and *interpretation* of XS and NS data because the structural and single-molecule length scales largely overlap [13]. Any experimental work with the acid-based systems faces

the additional challenges of handling the samples in completely anhydrous conditions, as water has been shown to affect dramatically the phase behavior. Of course, static studies will likely lead to dynamic ones such as rheo-SAXS, rheo-SANS, and rheo-optics (e.g., [14]).

Another key challenge compared to earlier work in the literature on polymeric and colloidal liquid crystals is the high absorbance of SWNT in the visible light range, which makes difficult optical microscopy; reflected light techniques will be helpful for studying the morphology of these mesophases (see ref. [15] for a recent application to MWNTs). Finally, possible future needs for online measurements and monitoring techniques should be considered while developing off-line (nano, micro, and meso) metrology tools for SWNT production and processing.

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INSTRUMENTATION AND METROLOGY AS KEY DRIVERS FOR ALL THE NNI GRAND CHALLENGES

Michael T. Postek, National Institute of Standards and Technology

Instrumentation and metrology (measurement science) are key components to the success of all the nine grand challenges of the National Nanotechnology Initiative. Metrology is pervasive and it is unifying. Instrumentation provides the necessary data on which scientific conclusions can be based, and correct metrology provides the ability to properly and accurately interpret those data.

The unifying effect of metrology is most epitomized for one of the more universal tools used in nanotechnology today, the scanning electron microscope (SEM). The SEM was initially evolved from an X-ray microanalytical tool into an imaging tool. The biologists were some of the first adopters of this technology, and a great deal of fundamental biological research was done with this instrument. In the mid-1980s, visionaries in the semiconductor industry saw the need of the application of SEMs in the inspection and metrology in semiconductor production because SEMs were already being used for research applications. Device and integrated circuit feature sizes were shrinking below the optical microscope capabilities achievable at that time. Low-accelerating voltage operation was chosen for its potential for nondestructive imaging and the fact that conductive coating was unnecessary to minimize sample charging [1-4]. Low-accelerating voltage operation also provided limited beam penetration into the sample and a more precise "look" at the surface structure. Imaging and information content was greatly different between high- and low-accelerating voltage operation, and a great deal of new knowledge about the samples resulted. The higher-brightness lanthanum hexaboride electron guns were being pushed to their limits, digital frame buffering was introduced, and eventually field emission instrumentation was introduced and became superior, proving their value of increased electron source brightness and higher resolution. All this took a tremendous amount of investment in research and development. Fortunately, the semiconductor industry was willing to pay for the development of special fully automated SEMs capable of meeting the needs of semiconductor production to view and measure nanometer-sized gate structures used on the modern semiconductor chips. In the meantime, this development benefited all other SEM applications for nanotechnology. This is especially true for biologists who have benefited in improved resolution and imaging capabilities for their nanometer-sized structures. This is a perfect example of cross-discipline needs and the interdigitation of technological solutions needed for commercialization of nanotechnology. This also points out that there is an immense cost for instrument research and development that must be born by some segment of the industry. In the case of the SEM, the semiconductor industry was already established and could bear that burden. Is there an emerging nanotechnology industry sector that can do the same for nanomanufacturing?

Measurements generally go hand in hand with ability to image. The need to measure nanometer-sized semiconductor structures is a similar need to the biologist or environmental nanometrologist who would like the size measurement of particles. Automated analysis for these purposes has yet to be fully developed. Precise metrology in the production environment is routine to the 1–5 nm level for semiconductor metrology, but accuracy remains lacking. To provide accurate measurements of any structure in the SEM, an integrated model of the electron beam/sample/instrument interactions must be developed. Such models have been developed for application to semiconductor manufacturing and employment of them in the measurement process has already improved the precision by a factor of 3 [5]. However, for lesser-known samples, especially those of biological nature, new measurement techniques need to be employed to obtain the necessary input data for a reliable model.

The SEM has another problem to overcome, which is charging. Another crossover technique that has been used in the biological and food research but that has just recently been adapted to semiconductor metrology

and inspection that minimizes, if not eliminates, the sample charging is environmental or high-pressure scanning electron microscopy. It offers the advantage and possible application of higher landing energies or accelerating voltages, different signal forming and contrast mechanisms and charge neutralization. This method employs a gaseous environment to help neutralize the charge build-up that occurs under irradiation with the electron beam. Although very desirable for the charge neutralization, for various technical reasons, this methodology has not been in common use in nanometrology for semiconductor inspection or metrology until just recently [6]. This is a relatively new application of this technology to this area and a good deal still needs to be learned. This technology shows great promise in the inspection, imaging and metrology of nanometer-sized structures on optical photomasks in a charge-free operational mode. In addition, this methodology affords a path that minimizes, if not potentially eliminates, the need for charge modeling that is needed for higher accuracy measurements. As stated earlier, the modeling of charging is exceptionally difficult since each sample, instrument, and operating mode can respond to charging in different ways. Therefore, this methodology, which effectively eliminates the charging, shows great potential if the optimal balance can be achieved in a reproducible manner. Further research needs to be undertaken to understand the ways to optimize the operating conditions for these instruments for nanotechnology research and metrology.

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METROLOGY FOR ATOMIC SCALE NANOFABRICATION

John Randall, Zyvex Corp.

Zyvex is committed to developing atomically precise nanofabrication and is therefore pleased to see this workshop focusing on metrology at this size scale, as it is certainly one of the key enabling technologies for this endeavor. Atomic-scale metrology presents some interesting challenges. The required resolution for metrology at the atomic level is subangstrom scale. There are exciting advances being reported that provide this resolution, and we are impressed with the work of Rick Silver here at NIST, which has demonstrated a variation of interferometry with subangstrom resolution.

We believe that this and related work on ultraprecision positional feedback is essential for atomic-scale nanofabriaction and related metrology. This belief is based on our opinion that, at this point in time, scanning probe-related technologies are the best method for detecting the position of individual atoms and molecules. Imaging alone by scanning probes is clearly insufficient for metrology, and therefore positional feedback is required. Nanopositioning technology must be developed to allow probes to interact with 3D surfaces, rather than the quasi-2D surfaces that are now scanned.

However, there is a serious limitation on the use of scanning probes for metrology. With current technology, the tip is an unknown structure (from the atomic level perspective), each tip must be carefully calibrated, and there are no atomically precise standards for this calibration. We should point out that a tip that provides good imaging for a scanning tunneling microscope does not meet our requirements. What is required is a technology that produces moderately high-aspect ratio tips with an atomically precise structure that is

invariant from tip to tip, and preferably with a single atom apex. The extent of the tip that must be known with atomic precision will depend on the metrology application, but to fully support atomic precision fabrication, it needs to extend at least several atomic layers away from the apex of the tip. Mechanical stiffness, electrical conductivity, and structural and chemical stability are all potentially important applications for most applications. A well-defined tip structure will be a huge advantage, not only to atomic-scale metrology but also to all scanning probe-based metrologies. Even with atomically precise tips, there is a need for atomically precise fiducial structures and size (line width) standards. Such standards would have an enormous impact on other metrology applications, such as those used in the semiconductor industry.

Atomically precise tips for metrology can be the starting point for atomically precise tools used for atomic and molecular manipulation. The invariant tip structure provides a stable platform from which to engineer specific functions on the end of the tip. This is key to atomically precise fabrication. For nanofabrication applications where the substrate is crystalline, there is an opportunity to use the atomic lattice as a fiducial grid. This would be an extension of the research conducted by Hank Smith's group at MIT on spatial phase locking, an approach that I think has significant merit.

A technology that I believe has huge promise is the 3D atom probe technology that has its roots in the most venerable (or at least the oldest) of atomic resolution imaging technologies, field ion microscopy. Imago Scientific Instruments has improved the efficiency of this technique and is able to deconstruct CMOS transistors, showing the distribution of individual dopant atoms. It is essentially an atomic resolution deconstruction technique that provides the position and species of the constituent atoms. Although it does have some limitations, such as being destructive, not detecting approximately 30% of the atoms, and needing relatively conductive samples, there are technical paths to improving the latter two limitations, and its capabilities are unmatched by any other technique. This will be a powerful tool for nanofabrication development efforts and is tailor-made for examining the structure of atomically precise metrology tips and atomic/molecular manipulation tools.

ABSTRACT FOR NNI WORKSHOP ON INSTRUMENTATION AND METROLOGY FOR NANOTECHNOLOGY

Jeff Rosner, Agilent Technologies

The perspective being brought is that of an industrial tool supplier to industry. Agilent has long been developing and supporting instruments for measurement and control and will increasingly play a role in delivering tools enabling nanotechnology research, development, and commercialization.

The metrology challenge can be discussed in three subdivisions: microelectronics, biotech, and bulk materials. In microelectronics, we will see dimensions substantially below 100 nm before 2010. Many measurement challenges will involve evolution of existing tools; ellipsometry, surface science techniques, and other R&D and yield management tools will evolve without heroic efforts. Molecular electronics will not be a reality before 2010; tools for this R&D effort will only be developed through intervention, as the market is not sufficient to fund the R&D required. Alternative modalities of scanning probe microscopy (SPM) will continue as the dominant change trends. In mainstream IC scaling, lithography, CD metrology and defect detection are the dominant unsolved problems. Lithography is well funded elsewhere; CD metrology by SEM will run out of steam soon and require alternative tools. Customized SPM tools are the most promising candidates today. Defect detection is moving from a mix of optical and ebeam tools to full use of ebeam tools, primarily SEM. For elemental work, X-ray spectroscopy on an SEM is still dominant, although field-emission Auger is being developed. High-resolution SEM/EDX at very low accelerating voltages is limited by detector resolution; microcalorimeters and other alternative approaches to the Si(Li) detector will make a large contribution. Large-area high-throughput SPM could be viable, but there are currently no approaches en route to commercialization. Calibration of SPM lateral and vertical scales is still an issue without a widely standardized solution.

There are tremendous opportunities in the biotech sphere for tool contributions. Many techniques used today are quite limited. Poised for revolution is optical spectroscopy, including fluorescent markers as well as visible demonstrations of vibrational techniques such as surface plasmon resonance (SPR) and surface-

enhanced Raman (SERS). Quantum dots and other nanoparticle markers offer multichannel replacements for organic dyes. This will require a new generation of instruments capable of hyperspectral operation (in contrast to the unique laser/filter pair for each channel required for organic dyes), with real-time multivariate analysis. Microscopes, scanners, and so on will all need development for high-throughput operation; high channel-count *in vivo* cellular imaging represents a huge opportunity to accelerate research in genomic and proteomic pathways. New SPM modalities are beginning to emerge, such as chemistry-specific force microscopy. Although many components exist, creating toolsets that are reproducible and sufficiently general purpose will require a substantial investment in applied research to understand the variability in current demonstrations.

Finally, the area of bulk materials represents a growing challenge with unknown solutions. Manufacturers of gram and kilogram quantities of both nanoparticles and composites are struggling with today's tools for microstructure examination. Although TEM has emerged as a dominant tool, it remains expensive, time consuming, and practiced exclusively by a few highly trained experts. Chemists and materials scientists in this field are reduced to stochastic observations to infer microstructure using bulk measures of spectroscopy, light scattering, SPM, voltammetry, and so forth. All these are severely limited in resolution, specificity, or sensitivity to some degree. The goal is to sample a statistically significant volume of material (number of particles or volume of composite) and know the full chemical constitution. Techniques like Imago's local electrode atom probe provide promise but are extremely limited in applicability. Revolutionary breakthroughs are needed, as many of the tools used today are mature and operating at their limits.

PERSPECTIVE IN NANOMETROLOGY

Ajit K. Roy, Air Force Research Laboratory

Continuous fiber-reinforced (6–20 μm in diameter) reinforced advanced composites have been in numerous structural applications for over three decades. Although advanced composites have offered many advantageous materials attributes over metallic materials, the materials discontinuity at the micrometer scale (the fiber matrix interface, in particular, causing stress concentration) of composites limits the performance potential of the material. For example, the origin of the process-induced residual stresses, interlaminar stresses at the lamina interfaces, and so on that limit composite strength, is essentially a result of the existence of the stress concentration of the fiber matrix interface. The enormous surface area-to-volume ratio of nanosize inclusions in composites offers a renewed opportunity of tailoring properties of composites to enhance performance, as well as adding functionality to its performance. The wide variety of size, shape, and properties of nanoinclusions, through innovation processing, may potentially offer a gradient microstructure to eliminate or reduce the order of material discontinuity that limits composite performance. One of the key challenges to making this achievable is to properly characterize the material morphology at appropriate scale, and at the nanometer scale in particular. For this we need to develop characterization tools and multiple measurement techniques to confirm accuracy of the measurements. Along with this we also need multiscale modeling to interactively assist developing the measurement tools/methodology and assess the nanotailoring effect to bulk response.

INSTRUMENTATION AND METROLOGY STRATEGIES FOR NANOFABRICATION

Gary W. Rubloff, University of Maryland

Nanotechnology is Dramatically Diversifying Microtechnology

The broad applications of nanotechnology have already made this apparent, where the functionality goals and materials/process approaches range from nanoelectronics to novel nanomaterials to biotechnology. More technically specific, the diversity of approaches and applications suggests a fundamental bifurcation in the way nanosystems are fabricated, specifically with respect to assembly and registration of nanostructures into nanosystems that perform useful functions.

Evolutionary path: The evolutionary path for nanotechnology is based on the fabrication of nanodevices as extrapolations of traditional materials and process approaches, as envisioned for transistor structures (e.g.,

vertical FET). Here spatial registration is achieved through improvements (or perhaps revolutions) in lithography, with increasing reliance on self-aligned structures controlled at the nanoscale. Some new processes may arise, such as nanoimprint lithography combined with high-selectivity processing; this could achieve nanoscale pattern definition in parallel processing more compatible with manufacturability. Evolutionary pathway will require profound advances in instrumentation and metrology. Characterization of vertical profiles, chemical analysis, and defect decoration will be critical. Scanning probe methods will be a leading player. Unusual approaches to metrology and testing are expected, such as techniques for decorating defects so that they can be characterized and repaired or removed.

Revolutionary paths: The astounding array of nanostructures (e.g., nanotubes, nanowires, biomolecules) opens the door to strikingly different scenarios for fabricating nanosystems, with applications from next-era computation to ubiquitous chemical and biological sensing. What distinguishes these from evolutionary pathways is radically different schemes for assembling functional systems. Assembly must be accomplished at both nanoscale and in relation to micro/macro scale. One can envision two scenarios for assembly of nanostructures. If the active nanostructure elements are self-assembled by reference to a template (e.g., 3D assembly as regular arrays of nanopores), then their assembly into circuits/systems presents what may be a nano-interconnect challenge. However, if the active nanostructure elements are not self-assembled, it is necessary to either custom connect them (given their randomized position and orientation) or transport, register, and align them as free objects. Schemes to deal with complex arrays of nanostructures, whether tethered to surfaces or freely movable in fluids, demand sensing, actuation, and control systems driven by metrology. This places a premium on the development of integrated scanning probes that are able to manage the assembly and/or interconnecting of nanostructures. Super-selective processes (e.g., biomolecule-based attachment) could be key enablers for intelligent immobilization of nanostructures where they need to be in order to assemble working circuits/systems.

10-Year Future for Nanometrology

These possibilities suggest an R&D emphasis on both evolutionary and revolutionary approaches. Here are some long-term research goals to consider:

- Embedded scanning probes or other test sites within MEMS environments to enable sensing, characterization, metrology, etc.
- Development of multilevel microfluidics, without and with active on-board microactuator control to broaden design capabilities
- Understanding and demonstration of biomolecular conjugation would make possible superselective attachment and assembly of various nanostructures (both biological and nanobased inorganic)
- Strategies for defect decoration (chemical, electrical, etc.), metrological sampling in vast arrays of nanostructures, and on-site repair (e.g., fuse-blowing) to substantially improve scale-up to functional nanosystems
- Identification and demonstration of approaches for microfluidic- and electrokinetic-based directed assembly of nanospecies to confirm viability of fluidics-based assembly of free nanostructural elements
- Understanding of what constitutes an efficient hierarchy for nanosystems assembly to clarify how nanoscale structures, nanoscale registration/connections, and microscale/macroscale inputs/outputs should be related to each other for system efficiency

GRAND CHALLENGE WORKSHOP ON NANOSCALE INSTRUMENTATION: ENVIRONMENTAL PERSPECTIVE

Nora Savage, U.S. Environmental Protection Agency

To successfully implement pollution prevention strategies and efficiently remediate currently contaminated environments, a detailed and accurate characterization of the environment must be performed. This requires knowledge of the various processes occurring at the nanoscale—within natural environments, between the natural environment and living systems, and between the different media within the environment. Specific

requirements for understanding the complex reactions, processes, and transformations that occur in the environment include computer modeling tools for predictions, methods for real-time, accurate observations, and techniques for manipulation and possibly alteration. A variety of instruments currently available provide limited information, but a more extensive set of tools is needed to improve the detail, depth, accuracy, and reliability of data.

Nanotechnology has the potential to provide this much-needed instrumentation. Within the next decade, methods for real-time detection and quantification of pollutant concentrations in a variety of environments, including subsurface, aqueous, and air environments, which are present at very low levels, should be achievable. In fact, current research has demonstrated the ability to detect one molecule of a substance. However, the challenge will be to develop innovative ways to get the molecule of interest to the detector when the substance of interest is present in very dilute concentrations. An additional challenge will be to successfully deploy the detector in hostile or difficult-to-access areas. The current evolution of sensors that are increasingly smaller, cheaper, more accurate, more sensitive, and increasingly flexible can address these issues by allowing for ubiquitous deployment of these devices in a variety of media and locations.

Achievements in nanotechnology also have the potential to deliver mechanisms that enable the study of nanoscale processes in the environment. Knowledge on the molecular level of the transformation, uptake, bioavailability, and biotransformation of compounds by animal and plant organisms as well as the transport of such substances to and from different media is essential. Such data will provide crucial information concerning the biological and ecological mechanisms by which pollutants are transferred, eliminated or reduced in living systems and ecological environments.

Key challenges for the advancement of environmental monitoring, detection, and analysis include the need for rapid, real-time, accurate data gathering and analysis techniques, and electronic transition of the data into user-friendly formats that provide meaningful information. In addition, the integration of data obtained using various methodologies, including geographic information systems (GIS), sensor elements and arrays, and models will provide a holistic view of the environment. Overcoming these environmental monitoring and detection challenges is critical for effective and sustained environmental protection.

THE METROLOGY CRISIS FOR NANOMANUFACTURING

Mark L. Schattenburg, Massachusetts Institute of Technology

The National Nanotechnology Initiative has spawned a great deal of new research and excitement about nanotechnology. Many pundits have predicted that nanomanufacturing on a wide scale is poised to take off and will revitalize the economy. However, a study of the last two industrial revolutions—the so-called machine tool revolution and the semiconductor revolution—reveals that a nanotechnology revolution is unlikely to happen anytime soon because of the serious lag of metrology technology and infrastructure.

As proof of this problem, one may simply glance at the promotional material of any leading nanotechnology conference or research journal and notice that typically half of the “images” of nanodevices are computer-generated simulations. This underscores the serious inadequacy of microscopy and metrology tools in the research environment, let alone in manufacturing. Anyone who has tried to image or measure sub-30 nm features using conventional electron beam or probe microscopes will understand the problem. Fuzzy, noisy images are par for the course. Sharp, low-noise images and measurements at the nanometer scale take Herculean efforts. This does not bode well for so-called nanomanufacturing, where rapid, accurate, high-resolution measurements are essential for profitability. In this presentation, the history of metrology in industrial revolutions will be reviewed. Suggested dimensional metrology infrastructure required for nanomanufacturing will be presented.

MANUFACTURE OF HIGH-ASPECT RATIO CARBON NANOTUBE ATOMIC FORCE MICROSCOPY PROBES

Y.N. Emirov, J.D. Schumacher, M. M. Beerbom, B. Lagel, B.B. Rossie, and R. Schlaf, University of South Florida

Carbon nanotubes (CNT) are promising candidates for high-aspect ratio atomic force microscopy (AFM) probes because of their exceptional mechanical properties. Applications in sub-100 nm critical dimension metrology are among the envisioned uses for such probes. The challenge for the preparation of CNT AFM probes lies in the precise placement of one well-defined CNT at the end of a regular Si cantilever tip fitting commercially available AFM equipment. This CNT needs to have a well-defined diameter, length and orientation, tailored for the structures to be characterized. We report about our recent progress in developing a manufacturing process for such CNT probes. Our process is based on CNT growth by plasma enhanced chemical vapor deposition (PECVD), which uses a catalyst to induce CNT growth. The need for a catalyst enables the specified placement and the definition of the diameter of the grown CNTs. Our method uses the focused ion beam (FIB) and electron beam lithography (EBL) techniques in combination with thin-film catalyst deposition techniques to define the catalyst patterns.

NUCLEAR MAGNETIC RESONANCE IMAGING USING MAGNETIC RESONANCE FORCE MICROSCOPY

Doran Smith, U.S. Army Research Laboratory

Magnetic resonance imagery (MRI) has had many benefits to medicine and biology. However, the low sensitivity of the conventional inductive detection of nuclear magnetic moments has limited MRI to the micrometer scale and above. The alternative technique of force detection of the magnetic resonance, magnetic resonance force microscopy (MRFM), is predicted to increase MRI sensitivity to one proton and the resolution to 1 Å. The force measured in a MRFM experiment is the force between two magnets: a small ferromagnet mounted on the end of a small cantilever, and the nuclear (or electron) magnetic moments of the sample. Until now conventional electrically detected MRI has lacked the sensitivity to make MRI of solids useful. (Note: medical MRI is liquids MRI, i.e., MRI of ugly bags of water, not solids MRI.) When perfected, MRFM is predicted to be able to image a single atom in a three-dimensional inhomogeneous object. Such ability will find broad applications in biology, medicine, materials science, semiconductors, polymers, and many other areas.

We apply force-detected magnetic resonance to the semiconductor GaAs, in combination with optical pumping to increase the nuclear spin polarization. We demonstrate one-dimensional nuclear magnetic resonance imaging of GaAs with 170 nm thick slices and resolve two regions of reduced nuclear spin polarization density separated by only 500 nm.

INSTRUMENTATION AND METROLOGY FOR NANOMANUFACTURING—INDUSTRY NEEDS

Sharon Smith, Lockheed Martin

Nanotechnology research and development and nanoprototype manufacturing are still in their infancy. The supporting instrumentation and metrology are just now evolving to support these areas. Although there will likely be carryover to full-scale manufacturing, we will need new, specialized metrology tools and processes for production in an industrial environment, and the scale-up between research and industrial applications will result in some new, unique, and very challenging problems where nanometrology will play a key role. Not only will there be the scientific problems to be answered, but there will also be economic, environmental, and perhaps societal and other implications to be addressed, all needed for us to realize the real potential of nanotechnology.

In the next 10 years, it should be possible to address a number of instrumentation and metrology requirements that will be needed for widespread, industrial nanomanufacturing. These will include new or

modified nanometrology tools, processes and techniques. Accuracy, precision, reliability, cost, time, and quality become very important parameters. Other life cycle effects (e.g., maintenance) should be considered. Other near-term considerations should include the ability to remotely access nanometrology tools, education requirements in nanometrology, and environmental concerns associated with nanomanufacturing and how nanometrology can help. More specific examples are provided in the accompanying two-chart presentation.

ESTABLISHING THE NEEDS OF CRITICAL ENVIRONMENTS IN FACILITIES SUPPORTING NANOTECHNOLOGY: THE SCIENCE BEHIND CONFLICT RESOLUTION

Ahmad Soueid, HDR Architecture, Inc.

As nanotechnology research compels the scientific world to explore new uncharted territories, scientists are increasingly demanding more stable research environments. Scientists are manipulating matter at the atomic and molecular scales to obtain materials and systems with significantly improved properties. As nanoscale research translates into nanofabrication and manufacturing, the physical environments allowing for both early research and development and later manufacturing operations impose more strenuous demands on facilities. These demands include high levels of accuracy in environmental criteria such as temperature and humidity control, vibration and acoustic isolation, air cleanliness from particulate matter, control of biological contaminants, electromagnetic interference (EMI), radio frequency interference (RFI), and good-quality electrical power.

The needs of facilities supporting nanoscale metrology will largely be influenced by (1) the effect of emerging technologies on measurement sciences (i.e., nanomaterials, nanoprobes, nanobiology, nanoelectronics, nanophysics, nanoscale building blocks, nanofluidics); (2) the measurement challenges presented by the emerging technologies; and (3) the techniques foreseen to meet those challenges (i.e., 3D characterization, dynamic measurement needs of nonlinear structures and organisms, and reducing the traceability chain in measurement standards). Many laboratory facilities are becoming obsolete to accommodate future research. Scientists are finding themselves spending time improving the physical environment and diverting resources from research. Increasingly stringent environmental criteria are demanding increasingly complex infrastructures. Institutions are realizing the need to renovate older facilities as well as designing/constructing new facilities with criteria that are more and more restrictive.

To be able to respond to the needs of nanoscale metrology, architects and engineers must be able to identify key design parameters and laboratory environmental requirements for measurement techniques. This is complicated by the fact that environmental requirements could be conflicting in their physical execution. (For instance, tight temperature control requires a higher level of air changes, which negatively affects the vibration and acoustical criteria.). A delicate balance of managing conflicting criteria is essential in the development of specialized facilities. Knowing which criterion is more important than another requires a detailed understanding of the functions within the space. While making comparative measurements against a particular standard, the control of fluctuations in temperature and humidity are more important than achieving an absolute accuracy of a temperature or humidity setpoints. The reverse may be true under different circumstances.

There are many available technical sessions/courses dealing with the design of different types of facilities; however, few deal with conflicts in criteria development. The Web site http://www.NANObuildings.com is a not-for-profit forum that was specifically created to deal with conflicts of such criteria and communicate them through a series of technical workshops called “Buildings for Advanced Technology Workshop” (BAT). Such workshops are a way to communicate ideas, solutions and lessons learned on other similar projects between scientists, users and different design teams. Project-specific workshops are valuable because the users must communicate to the designers the specifics of the criteria (i.e., magnitude, limits on time variation, limits on spatial variation, etc.) as well as the relative importance of the different criteria (to aid in the resolution of criterion conflict). Designers must communicate to the builders the specifics for interpretation of criteria, as well as detailed means for evaluation (i.e., spatial averages, time averages, maxima over time or space). If communication is adequately carried out, the probability of achieving the ideal environment is greatly enhanced.

METROLOGY FOR MOLECULAR ELECTRONICS

Duncan Stewart, Hewlett-Packard Laboratories

The ultimate limit of integrated circuit scaling frequently has been proposed to be a single-molecule active device, potentially on the order of ~1 nm in size. Hewlett-Packard Labs initiated a dedicated effort in this field of molecular electronics 3 years ago. We have focused significant effort on a planar metal/molecular monolayer/metal cross point device structure where the molecules act as active or passive two-terminal devices. At each step of device fabrication and characterization new metrology needs have been encountered, the most difficult of which relate to the incorporation of nanometer-thick organic films on top and inside of inorganic device structures. This is a research area with essentially no standard metrology techniques. The most productive characterizations to date have been achieved by applying new combinations of chemistry and surface science methods, with both large area probes and localized SPM methods. I will use our work on the metal/organic monolayer/metal devices as a case study of these new metrology challenges, relevant to both molecular electronics and most nanostructured chemical and biological sensor devices.

INSTRUMENTING MECHANICAL MEASUREMENTS AT THE NANOMETER SCALE

Chanmin Su, Veeco Instruments

Scanning probe-based measurement technology represents one of the fastest growing areas of instrumentation. Based on ISI Web of Science (formerly Science Citation Index) the papers published with subjects related to atomic force microscopy have grown by over two orders of magnitude since 1989 to about 4000 journal papers last year. Scanning Probe Microscopy (SPM) is an important platform for nanotechnology in imaging, manipulation and characterization of materials. In metrology SPM has already played a successful and unique role in dimensional measurements, providing high-resolution 3D profiles, side-wall measurements without which semiconductor industry development would be hindered. Given that the probe interacts mechanically with materials at the nanometer scale, it is natural to extend SPM's contribution to quantitative mechanical property characterization. Non-AFM and AFM based platforms are about equally represented. The characteristics of each method are listed in the table below.

Method	Mechanical Properties	Spatial Resolution	Force Reso.	Frequency Bandwidth	Quanti. Meas.	Primary Measurement Accuracy Factors	Other Factors
Nanoindentation	Modulus Viscoelasticity Hardness	sub-µm	nN	300 Hz	Yes	Contact Geometry	1. Force resolution 2. Soft materials, mechanical standards from 100kPa to 10 GPa
Nanoindentation AFM	Modulus Viscoelasticity Hardness	nm	pN	kHz Discontinuous	No	1. Contact geometry (order of magnitude) 2. Application of force 3. Force Calibration	Soft materials, mechanical standards from 100kPa to 10 GPa
Lateral force AFM	Friction	nm	nN	DC Discontinuous	No	1. Contact geometry 2. Lateral force calibration 3. Lateral force standard	Mechanical standard testing and analysis algorithm
Tapping Mode AFM	Modulus Viscoelasticity	nm	pN	100s kHz Discontinuous	No	1. Complicated interaction 2. Excitation force in fluid	Mechanical standard
Torsion Mode AFM	Dynamic friction Viscoelasticity	nm	pN	Up to 1 MHz	No	Physics of near field shear interaction	Mechanical standard
AFM Force Spectroscopy	Intermolecular binding force	Single molecule	pN	30 Hz	Yes/No	1. Non-specific interaction 2. Intermolecular force metrology standard	Thermal noise
Ultrasonic AFM	Modulus Viscoelasticity	nm	pN	MHz	No	1. Contact geometry 2. Ultrasonic source/impedance	Mechanical standard
Force Modulation AFM	Modulus Viscoelasticity	nm	pN	kHz	No	1. Contact Geometry 2. Force application source	Mechanical standard

As is seen in the table, the AFM platform has the desired resolution in terms of measurement volume and force scales. However, whenever a tip engages in mechanical contact with a sample, the contact geometry presents the largest inconsistency and measurement error source in analyzing mechanical properties.

Nanoindentation tools minimize the problem by using a standard diamond tip. Even then, a well-prepared surface for consistent contact is an important factor. We explore each method listed in the table and illustrate how its quantitative mechanical metrology is impaired. Solutions that address fabrication and cost issues related to the following problems are critical for U.S. industry to advance toward nanometer scale mechanical metrology: contact geometry characterization, force calibration, and force application source and bandwidth.

NANOMETROLOGY FOR NEW SPINTRONICS

Mark Tondra, NVE Corp.

The commercial driving forces of nanomagnetism are the hard disk drive industry, other discrete magnetic storage modes, and the magnetoresistive random access memory (MRAM) development effort. The present track width on hard disks is now less than the feature size of standard silicon foundries (50 nm vs. 90 nm). The shrinking of hard disk bit size at the current rate of 100%/year will ensure that these efforts continue to lead the way in terms of smallest featured fabrication in magnetics. MRAM is at the stage of rapid ramp-up to commercial production. It is expected that the bit density will approach the smallest feature size of standard silicon devices in the next five years or so.

Taking these two data storage applications to be the defining problems for the next decade, and using the present rate of density increase as a means of projection, one can see that commercial devices will have dimensions on the order of 1–10 nm in the year 2014. Some key issues for hard disk media are, first, the magnetic grain size will need to be less than 10 nm, and be controllable on this length scale. The introduction of patterned media in commercial drives is a distinct possibility. Second, more sophisticated structures are needed (perpendicular media, AF-coupled media, etc.) to keep the density ramping up. For hard disk read heads and MRAM, the challenges are in understanding the behavior of magnetoresistive devices on the 10 nm length scale. Specifically, there are issues of curling, vortex, layer–layer coupling, effects of defects and roughness, interfaces, temperature, and so on. In all cases, the relevant timescale will shrink below 1 ns. For MRAM in particular, reducing the energy required to switch and read bits will mean better control of switching sequences on the 100-ps time frame.

The need for new Spintronics devices in these industries will occur when the means of storing and reading information are not viable at the relevant dimensions. The most immediate hurdle appears to be the thermal stability of magnetic objects on the 10 nm scale. There are several development efforts to incorporate thermal action in the writing procedures. If these are successful, it is likely that the present modes of devices can be extended to the 10 nm scale. At some point around 10 nm, new spintronics devices may become attractive if they can be designed to have stable states at room temperature. Some promising new areas are: spin momentum transfer, spin-wall interactions, internal spin structure manipulation, spin packets, time-dependent phenomena, high-frequency magnetic excitations, quantized conductance, and magneto-thermal effects.

With this technology path in mind, here is a proposed nanometrology challenge:

- Measure the magnetic field with 0.25 nm spatial resolution (on-wafer if possible)
- Measure moment of 100 × 100 × 100 atoms
- Predict internal moment orientation structure/s
- Detect using scanning instrument
- Measure these properties on 10 ps timescale

As the hard disk industry and MRAM make 100 nm sized features mundane over the next 10 years, new "spinoff" applications of nanomagnetism are likely to spring up. The most promising areas appear to be in nanomachinery (pumps, gears, motors, etc.) and biological devices (biosensors, implanted electronics, artificial cells). The nanometrology tools developed for the data storage industry will likely be applied to a growing and fascinating field of new nanodevices. This, in turn, could inspire a new set of metrology requirements.

AN INDUSTRY PERSPECTIVE ON NEEDS FOR INSTRUMENTATION AND METROLOGY STANDARDS FOR NANOTECHNOLOGY

Jonathan L. Tucker, Keithley Instruments, Inc.

Electrical measurements provide the underpinning for many nanotechnology discoveries. Instrumentation suppliers must continue to develop new techniques and equipment to support cutting edge research. All measurement equipment or tools used for nanoscale measurements need metrology standards and measurement protocols so that repeatable and verifiable measurements can be performed.

Historically, many scientific advances occur only after suitable investigative instruments become available. Today, tools such as the atomic force microscope (AFM), the scanning electron microscope (SEM), and semiconductor characterization systems help nanotech researchers visualize, resolve, and perform electrical characterization of nanoscale objects and devices. The information obtained with these tools allows researchers to manipulate atoms and molecules to create new materials, structures, and electronics. But for electrical measurements, we need to ask ourselves whether or not volts, ohms, and amps mean the same thing on the nanoscale as they do on a macroscale. Assuming Ohm's Law does not mean the same thing on the nanoscale, to what references do we compare any measurement? How do we measure them accurately and precisely?

In 2002, the NNI expanded the grand challenges to include more sophisticated and standardized nanoscale instrumentation and metrology designed to provide higher performance and measurement efficiency at lower cost. The NNI committee outlined instruments and tools for measurement, manipulation, and analysis that will not only support current activity, but also take nanotechnology to the next level. But without metrology standards, how do instrumentation suppliers create the next generation of tools without a means to compare results and verify performance?

Meeting the grand challenge of developing instrumentation and metrology standards must be a cooperative effort between instrumentation suppliers, user organizations, standards organizations, industry, and academia. Organizations such as NIST and other worldwide bodies, working with many industry partners, must develop the next generation of instruments and standards so that instrumentation has traceability to a recognized set of standards. We must develop measurement methods and protocols, and testing structures that allow for repeatable and verifiable data. Once these are in place, commercial companies such as instrumentation vendors can supply next-generation tools. Startup companies and large corporations will be able to perform incoming inspection so they can be assured that the nanomaterials they purchase are exactly what they are purchasing. Next-generation electronics can be manufactured and tested to meet the demands of consumer electronics.

Many challenges lie ahead. Time is critical. We need to know what we are measuring. Advanced microscopes, probes, and measurement workstations have opened up a whole new world with many potentials and promises. By working together, we can develop knowledge that will create solutions to the many problems that are core issues in the development of novel materials and electrical components. It is imperative that measurement standards, protocols, structures, and reference tools be developed and agreed upon in order to move forward. This will allow commercial manufacturing and production to take place. And it will be the new the products from nanotechnology research that fuel the economy of the world. But only until we have agreed upon metrology standards and methodologies can we then see the economic effect that has been projected.

METROLOGY FOR INTEGRATED CIRCUIT NANOMANUFACTURING

Vladimir Ukraintsev, Texas Instruments, Inc.

Superiority in integrated circuit (IC) manufacturing, the foundation for modern information and communication technologies, is critical for prosperity of our nation. Maintaining our current leading position in IC manufacturing is pivotal for establishing future success in nanotechnology. Several semiconductor companies are developing so-called "45 nm technology node" at this time. The 45 nm technology node (as

defined by the ITRS) falls in the domain of nanotechnologies. Therefore, the ITRS metrology requirements for the 45 nm node are considered *current nanotechnology needs*.

IC manufacturing metrology needs a boost. Both development and even manufacturing of the latest IC technology nodes (130, 90, 65, and 45 nm) suffer from deficient metrology. Critical "in-die" dimension (CD) metrology as provided by SEM is insufficient, because SEM has large sample-to-sample bias variation and, therefore, poor total measurement uncertainty. Scatterometry, however, does not monitor "in-die" CDs. The greatest needs for today's IC metrology are therefore:

- High-resolution "in-die" three-dimensional (3D) CD metrology with total measurement uncertainty (TMU) < 1 nm (true bottom CD, line and trench profiles, high-frequency line edge roughness)
- Physical and electrical metrology of complex material stacks (concentration, porosity, interfacial defects, stress, carrier mobility, etc.)

To define the *future metrology needs* of nanomanufacturing we make the following assumptions. First, we believe that lack of robust and inexpensive manufacturing solutions will be a show-stopper for novel nanotechnology. This contributes to the expectation that FET-based processes will remain the primary IC technology for the next 10–15 years. Second, we expect that lithography will remain a primary patterning technique. Elements of patterning by self-assembly could be used to arrange an array of repeatable cells (e.g., memory cells). Finally, we think that the next most probable technology step would consist of hybrid ICs with elements of charge-based nanodevices connected to the outside world using CMOS technology. Based on these assumptions we expect that the following metrology needs would be critical for future IC nanomanufacturing:

- Nondestructive 3D tomography with atomic-resolution capable of CD metrology and elemental analysis
- High-resolution physical and electrical characterization of interfaces (composition, atomic structure, defects, traps, energy structure, charge, stress, mobility, etc.)

We have *great challenges* ahead of us. Simple improvements of existing techniques will not be adequate. We need breakthroughs on several fronts:

- Nondestructive "in-die" 3D microscopy with atomic resolution. Probing at the atomic scale is a challenge. A breakthrough is needed: novel SPM, X-ray scattering, e-holography, etc.
- Nondestructive single atom detection, with chemical and electrical state characterization. Ultra-high signal-to-noise detection is a challenge. A breakthrough in spectroscopy is needed: EDS, EELS, NMR, RBS, etc.

It is uncertain whether circuit designers will find solutions to compensate for insufficient metrology and, by implication, process control, or whether we will witness breakthroughs in metrology and characterization. However, one way or another, progress will happen.

SOME DIMENSIONAL METROLOGY ISSUES FOR NANOTECHNOLOGY

John Villarrubia, National Institute of Standards and Technology

The Need for Nanometer-Scale Dimensional Metrology

The "nano" in "nanotechnology" refers to the nanometer size scale. A particular technology is legitimately classified as a nanotechnology because its function is inextricably tied to the properties of materials at that size scale. Where component sizes are themselves measured in nanometers, a change of a few nanometers represents a significant percentage difference, and therefore likely a significant difference in properties. For good process control, measurement uncertainty must be small compared to the size of these changes. With gate sizes still larger than 50 nm, the semiconductor industry already desires metrology with accuracies of 1 nm or better [1]. The requirements for trouble-shooting and process control of other nanotechnology industries are likely to be at least as strict.

Image Artifacts and Dimensional Metrology

Dimensions of very small objects are most commonly determined by measuring an image acquired by a microscope and applying a correction for the scale (i.e., magnification). Images are representations of the sample, but they are not perfect. Apart from the different scale, they are subject to a number of image "artifacts." For example, geometrical distortions may be caused by lens imperfections or in scanning microscopies like scanning electron microscopy (SEM) or atomic force microscopy (AFM) by scanner nonlinearities. Because of this artifact, the scale may be different in different parts of the image, and distances that are identical on the image may be different in the actual sample.

Another artifact is a consequence of a microscope's limited spatial resolution. Point features appear to have finite extent when imaged. Even when the image scale is everywhere correct so objects' center-to-center distances are accurately represented, their measured widths are distorted by this effect. For larger objects, the spatial resolution is small compared to features of interest, and this artifact is hardly noticed. For nanometer-scale objects, this is no longer the case. There are important, often nonlinear, effects that depend on the complexities of the interaction between the microscope "probe" (electron beam in the SEM, mechanical tip in the AFM) and the sample. AFM images are formed by scanning the sample with a mechanical tip that is either in contact or near contact. This results in an image that is a dilation of the sample and tip shapes [3]. Positive features (protrusions) on the sample appear wider by an amount that depends upon the dimensions of the tip and the heights and slopes of the tip and feature. Negative features (depressions) on the sample appear narrower by an amount determined similarly. Under favorable circumstances even a relatively blunt tip can yield atomic resolution because only a very small part of it ever interacts with the sample. More generally, however, for samples with larger slopes and higher aspect ratio features, artifacts comparable to the size of the tip are to be expected.

For secondary electron imaging in the SEM, incident electrons scatter inside the sample within some interaction volume. Secondary electrons produced deep inside the sample do not contribute to the image because they do not have enough energy to escape the sample. Only secondary electrons generated near a surface can contribute. The interaction volume intersects a greater part of sloped surfaces than horizontal ones, causing such surfaces to be brighter. Whenever the interaction volume intersects a new surface of the sample, new escape routes for secondary electrons can contribute to the image. For example, this can happen when the landing spot for the incident electron beam approaches a corner, and secondary electrons may escape from the side as well as the top surface. These nonlinear effects help to create the contrast pattern that defines the image and are also important for critical nanotechnology features.

An Approach to Addressing the Issues

Microscopes, as discussed in the introduction, always have image artifacts at some scale. Historically, when that scale became limiting, better microscopies with still smaller limits were employed. The same strategy will still be effective to a certain extent, but the nanometer scale is approaching the limits of many kinds of interactions between probes and materials. It will consequently be more important to understand these interactions, model them, and apply corrections. In the AFM, if the tip shape can be measured, those parts of the sample that were probed by the tip may be recovered from the image [2, 3]. In the SEM, modeling of the interaction between the electron beam and the sample can permit location of an edge with an accuracy better than the instrument's resolution [4, 5]. The situation is rendered more complicated in the incipient nanotechnology industries by the geometrical complexity of some of the envisioned parts to be manufactured. Existing models for the AFM require parts to be described as single-valued functions, $f(x,y)$.

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MAGNETIC NANOTECHNOLOGY AND METROLOGY NEEDS IN MAGNETIC RECORDING

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The areal density in magnetic recording has surpassed ~70 Gbit/in^2 in products and ~110 Gbit/in^2 in laboratory demonstrations. At 100 Gbit/in^2, the bit dimensions are about 35 nm (bit length) and 200 nm (track pitch, bit width) translating into data rates beyond Gbit/s for high-end drives (15,000 rpm, 2.5 inch dia disk; max. linear velocity = 50 m/s). The media are composed of Co-alloy granular materials with horizontal orientation of the magnetization (longitudinal recording). Recording heads are based on inductive writers with pole moment densities near 2.4 T and giant magneto-resistance field sensors with magnetoresistance values near 20% (CIP-giant magnetoresistance spin valve). Media grain sizes are 8–10 nm, and grain size distributions are near 20% (sigma over mean). Going beyond 100 Gbit/in^2 requires magnetically harder materials with smaller, thermally stable grains (5–8 nm) and tighter distributions (<15%). Experiments indicate that this may be possible in perpendicular recording, where a soft magnetic underlayer is used to enhance the write field, enabling such grains to be recorded on. Basic technology demonstrations of 170 Gbit/in^2 have been reported, and modeling suggests that extensions to about 1 Tbit/in^2 are possible using that technology.

A number of unique magnetic metrology needs arise because of the perpendicular magnetization orientation and the presence of a soft magnetic underlayer in the media. Besides the general need for larger fields in magnetometry (media anisotropy fields > 2 T) there is a need to decouple hard from soft materials, favoring magneto-optical methods. It is also necessary to correct for demagnetization effects and to characterize magnetic dispersions owing to segregation variations and to variations in the exchange coupling from grain to grain. Practical magnetic imaging on the sub-10 nm length scale is critical to improve the microscopic understanding of bit transitions, which not only depend on media nanostructural parameters (grain size, dispersions, exchange, cluster size) but also on the recording process (write field gradient, affecting media transition parameter). An important challenge is to directly measure and map the head field at the relevant timescale of 100 ps to 1 ns. At such speeds, precessional effects dominate and real-time, pump probe techniques are being explored. Angled fields can reduce the switching coercivity by up to a factor of two (45 degrees, Stoner Wohlfarth switching astroid), and several novel write head designs (trailing pole, Mallary head) and media designs (tilted anisotropy media, Gao and Bertram) have been proposed. Most of these designs are based on micromagnetic model calculations, which need to be founded in experiments.

Going much beyond Tbit/in^2 will require even more drastic changes of heads and media. One of the fundamental limitations relates to the media sputter fabrication process, which may not allow the tight grain size and magnetic dispersions required in models. Self-organized magnetic arrays (SOMA) of chemically synthesized FePt nanoparticles are therefore being explored as alternatives. These structures do not only show extremely tight size distributions (<5%) but are also magnetically much harder than current Co alloys. Key challenges are control of the magnetic easy axis, avoidance of sintering in FePt (annealing requirement of about 700°C can lead to grain growth) and establishing large-scale ordering and registry on the length scale of a disc. In addition, writing will require temporal heating and cooling in a magnetic field (HAMR: heat-assisted magnetic recording), which sets the stage of a whole host of new needs in heads (heat and field delivery schemes at sub-25 nm dimensions), media (thermal property control, temperature-dependent magnetics, Curie temperatures, blocking temperatures, etc.) and head disc interface (e.g., heat-resistant new lubricants and wear/corrosion-resistant materials). It is envisioned, that eventually a combination of SOMA and HAMR may lead to single particle per bit recording, with ultimate densities near 50 Tbit/in^2 (10 years

storage time, ambient temperature, FePt type anisotropies). Some form of assisted self-assembly, for example, via topographic or chemical prepatterning on the micrometer length scale, would be required to coat discs with the required uniformity. A single particle (bit) in this scenario has lateral dimensions of about 3 nm, corresponding to the single-particle superparamagnetic limit for FePt and comprises only about 1000 atoms, with a large fraction of atoms occupying the surface. It will be critical to develop magnetic measurements and structural tools to characterize such small magnetic units and their surfaces, which may be quite different from bulk and be strongly dependent on the chemical environment.

SPM-BASED METROLOGY

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As staff member of the PTB, the German National Metrology Institute, I have been involved in nano- and micrometrology for about 15 years. My current interest is concentrated on the use of scanning probe microscopes (SPM) for the traceable measurement of dimensional and related quantities. In addition, of course, other means such as X-ray reflectometry, ellipsometry, and interference microscopy are also being used for calibration purposes. Advances in all these methods are necessary. Here, I want to concentrate on SPMs, as I am convinced that this principle still has great potential for nanometrology.

In industry and science SPMs are widely used for research purposes. But industrial metrology cannot be imagined without these microscopes either. As soon as they are incorporated in a quality system, the correctness of the measurements becomes a matter of importance. The manufacturers meanwhile offer equipment allowing for this aspect and use hardware and software solutions to improve or even avoid the well-known disadvantageous properties of piezo scanners such as hysteresis, creep, and drift. National Metrology Institutes have set up reference devices that allow measurements traceable to the SI unit and are able to state measurement uncertainty figures.

Basically, the measurement tasks we are confronted with are width, height, distance, texture/roughness, thickness of layer, and hardness. The objects are nanotechnology products or calibration artifacts that very often are produced by microtechnology processes—the typical top-down approach. And thus also the measurement approach is a scaling down of methods known from the micro/macro world.

The results of a number of comparison measurements, including international key comparisons show that the uncertainty figures achieved by SPMs and other suitable instruments are in the range of a nanometer for pitch measurements and of several tenths of nanometers for step height and thickness of layer measurements. Measurement uncertainty figures are increasing considerably where shape and probe-sample interaction comes into play, such as with width measurements.

Short-Term Needs

The main obstacles to improved measurement uncertainties are the lack of knowledge of the actual interaction between probe and sample, and the effective shape of the probe. Once this problem is tackled and the uncertainty contribution of tip shape and interaction is lowered, the improvement of positioning accuracy is sensible and likewise also the calibration accuracy. The use of the crystalline lattice as a scale could then be advantageous (X-ray interferometry), and standards based on the crystalline lattice will then be needed. Some other problems, such as restricted measurement range and measurement speed, are being worked on. For instance, a so-called nano measuring machine has been developed and is commercially available (measuring range, 25 mm × 25 mm × 5 mm).

Medium-Term Needs

Scanning force microscopes have high potential for all sorts of analytics, provided the force can be measured with sufficient accuracy. Current methods allow force calibrations with uncertainties of tens of percents—much too high for meaningful measurements. A fast and simple method for traceable force calibration is needed.

Long-Term Needs

One of the principle disadvantages of current SPM designs is the unfavorable ratio of instrument volume to measurement volume. This gives rise to severe problems, such as Abbe errors, drift, and restricted measurement speed. A miniaturization of the scanning apparatus and of the displacement sensors could reduce these contributions to uncertainty and measurement time. True three-dimensional measurement capabilities are needed. The current cantilever principle is not suitable for that purpose. Perhaps, the optical levitation principle can be used for measurement purposes.

NANOCHARACTERIZATION CHALLENGE: STANDARD METHOD FOR ASSESSMENT OF NANOTUBE MATERIAL QUALITY

Leonard Yowell and Sivaram Arepalli, NASA Johnson Space Center

The surge of interest in single-wall carbon nanotubes (SWCNT) and their applications has stretched the limits of nanotube production capacity as well as materials characterization techniques. Researchers from industry, academia, and government laboratories have been using SWCNTs from the full range of available production methods. These methods result in substantially different tubes (diameter, length, etc.) as well as different amounts and types of impurities (metals, amorphous and graphitic carbon). Because of the presence of impurities in the raw product, most researchers would prefer to use purified nanotubes of the highest reasonable quality.

In the course of our work at NASA, it has become necessary to develop a standard characterization protocol for the evaluation of our material and the validation of our purification methods. There is no single analytical technique that can characterize the essential elements of sample quality: purity, homogeneity, thermal stability, and dispersability. We have performed a systematic evaluation of available characterization techniques and evaluated their use in analyzing our material. These standard analytical techniques include scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetric analysis (TGA), Raman, and UV-VIS-NIR spectrometry. Our suggested protocol standardizes measurements using these established techniques, and consumes <30 mg of material. Images from SEM are used to give a rough qualitative assessment of material quality. Higher-resolution images from TEM are used primarily to monitor the surface texture of individual ropes and to establish diameter distribution of the tubes in the sample. We currently lack an efficient method for determining the length distribution of individual SWCNTs. Raman spectra are used to estimate the extent of amorphous carbon as well as damage to the tubes. Analysis of TGA data is used to quantify the quality of the tubes (decomposition temperature) as well as the extent of noncarbon impurities in the collected sample. Absorption spectra of nanotube solutions are obtained by using a UV-VIS-NIR spectrometer and the variation of optical density with time is used as a measure of SWCNT dispersability. Dispersion, and the characterization thereof, seems at present to be the single most critical issue in developing SWCNT composites.

The establishment and community acceptance of a standard SWCNT characterization protocol is necessary in the development of reliable, high-performance nanotube-based materials for a wide range of applications. As a first step toward this goal, a workshop organized jointly by the National Aeronautics and Space Administration, Lyndon B. Johnson Space Center (NASA/JSC), and the National Institute of Standards and Technology (NIST) was held on May 27–29, 2003, at NIST in Gaithersburg, Maryland, to discuss and prioritize measurement needs relative to SWCNT purity and dispersion. A follow-on workshop was held in Gaithersburg, MD on January 26-28, 2005 by the same organizers.

OVERVIEW OF NANOTECHNOLOGY RESEARCH AT NASA GLENN RESEARCH CENTER

Mary Zeller, NASA Glenn Research Center

NASA Glenn Research Center is performing research in high-temperature nanotechnology for harsh environment aerospace applications. Three areas important for NASA missions are being addressed: materials, instrumentation, and power. During this presentation the various tasks that are currently being

investigated will be described. The innovative technology, the research approach and the accomplishments to date will be discussed.

In the materials areas, work is being done in self-assembly supramolecular materials. These polymer structures may have unique mechanical properties to exploit in synthesizing high-strength, high-toughness composites. These nanomaterials may also act as a template to attach groups with special electrical or photonic properties for possible applications as sensors or electrodes for batteries or proton membranes for fuel cells. Other researchers investigating novel materials are synthesizing materials for high-temperature operation. Clay and metal oxide nanoparticles; nano-onions; and carbon, boron nitride, and silicon carbide nanotubes are the materials being developed for high-performing composites, nanosensors, space lubricants; energy storage; and NEMS (nanoelectromechanical systems).

The instrumentation and sensors researchers are investigating quantum optics for communication and sensing application for both aeronautics and space environments. Currently the quantum entanglement (QE) apparatus that includes a novel high-speed photon counting detection system has been demonstrated to send data signals in a fraction of the time required for the state-of-the-art QE systems. Nanophotonics apparatus for control of nanoarrays has been designed, assembled, and operated with dual beams to trap and levitate micrometer sized particles not only in solvent but also in air. A vacuum chamber is being added to the laser tweezers system to enable trapping and control of nanoarrays of irregular objects such as nanotubes. This instrument is being designed to assist in nanofabrication for nanoelectronic devices. NASA researchers are also working in the area of power generation and energy storage. They are working on designing and testing power and energy storage devices using quantum dots, nanotubes, and so on.

Appendix D. Glossary

Ω	ohm
2D	Two-dimensional
3D	Three-dimensional
10×	factor of 10

A

A	Ampere
Am^2	Ampere-square meter
AAIA	American Institute of Aeronautics and Astronautics
ADF	Air-dynamic forming
AEM	Analytic Electron Microscope
AFM	Atomic Force Microscope
Ag	Silver
AGM	Alternating gradient magnetometer
As	Arsenic
AML	Advanced Measurement Laboratory
ASCI	Accelerated Strategic Computing Initiative
ASME	American Society of Mechanical Engineers
Au	Gold

B

BARC	Bead array counter

C

CD	Circular dichroism
CD	Critical dimension
CD SEM	Critical dimension scanning electron microscope
CIP-GMR	Current in-film plane, giant magnetoresistance
CMOS	complementary metal oxide semiconductor
Co	Cobalt
CRM	Certified reference material
C-V	Capacitance-voltage

D

DARPA	Defense Advanced Research Projects Agency
DHS	Department of Homeland Security
DNA	Deoxyribonucleic acid
DOD	U.S. Department of Defense
DOE	U.S. Department of Energy

E

EDS	Energy dispersive X-ray spectrometry
EDX	Energy dispersive X-ray
EELS	Electron energy loss spectroscopy
EFTEM	Energy filtered transmission electron microscopy
EM	Electron microscopy
EMI	Electro magnetic interference
EMU	electromagnetic unit
EPA	Environmental Protection Agency
EPIC	Electronic and photonic integrated circuit
EPMA	Electron probe microanalyzers
eV	Electron volt

F

fA	Femtoampere
FCC	Face-centered cubic
FDA	Food and Drug Administration
Fe	Iron
FET	Field effect transistor
FIB	Focused Ion Beam
FIM	Field ion microscope
FinFET	Fin field effect transistor
Flops	Floating-point operations per second
FMR	Ferromagnetic resonance
FTIR	Fourier transform infrared

G

Ga	Gallium
Ge	Germanium
GIF	Gatan's imaging filter
GHz	Gigahertz = 10^9 hertz
Gbit (Gb)	Gigabit = 10^9 bits
GMR	Giant magnetoresistance

H

H	Magnetic field strength
H_c	Coercivity or coercive (magnetic) field strength
HAMR	Heat-assisted magnetic recording
HEDM	High energy density material
HTREM	High-resolution transmission electron microscopy
Hz	Hertz, a unit of frequency equal to one cycle per second

I

IC	Integrated circuit
IIT	Instrumented indentation testing
in^2	Square inch
IR	Infrared
ISS	Ion scattering spectroscopy
ITRS	International Technology Roadmap for Semiconductors
I-V	Current-voltage

K

K, K_u	Magnetic anisotropy of a material
K	Kelvin, measurement of temperature
kA/m	Kiloamperes per meter
k_B	Boltzmann constant

L

$L1_0$	crystal structure type (strukturbericht designation)
LEAP	Local electrode atom probe
LED	Light-emitting diode
LMMS	Laser microprobe mass spectrometry

M

M (m)	Magnetization (magnetic moment)
M_s	Magnetization
mA	Millamperes
mag-lev	Magnetic levitation
MD	Molecular dynamics
MEMS	Microelectromechanical systems
MFM	Magnetic force microscopy
MIT	Massachusetts Institute of Technology
MOIF	Magneto-optic indicator film

MOSFET	Metal-oxide-silicon field effect transistor
MRAM	Magnetic random access memory
MRI	Magnetic resonance imaging

N

NA	Numerical aperture
NAIL	Numerical aperture increasing lens
NASA	National Aeronautics and Space Administration
NDE	Nondestructive evaluation
NDP	Nano disperse powder
NEMS	Nanoelectromechanical systems
NIH	National Institutes of Health
NIRT	Nanoscale Interdisciplinary Research Teams
NIST	National Institute of Standards and Technology
nm	Nanometer
nm^3	Cubic nanometer
NMR	Nuclear magnetic resonance (spectroscopy)
NNCO	National Nanotechnology Coordination Office
NNI	National Nanotechnology Initiative
NP	Nanoparticles
ns	Nanosecond
NSF	National Science Foundation
NSEC	Nanoscale Science and Engineering Center (NSF funded)
NSET	Nanoscale Science, Engineering, and Technology (NSTC subcommittee)
NSOM	Near-field scanning optical microscopy
NSTC	National Science and Technology Council
NT	Nanotubes
NW	Nanowires

O

ONR	Office of Naval Research
ORNL	Oak Ridge National Laboratory

P

p	Pico = 10^{-12}
Pa	Pascal, unit of pressure
PCAST	President's Council of Advisors on Science and Technology
PCR	Polymerase chain reaction
PEELS	Parallel electron energy loss spectrophotometer
PGNP	Prompt gamma neutron profiling
PIXE	Proton-induced X-ray emission
pm	Picometer
ps	Picosecond
PSE	Problem-solving environments
Pt	Platinum

Q

QD	Quantum dot

R

RBS	Rutherford backscattering spectrometry
RF	Radio frequency
RTD	Resonant tunneling diode

S

SAXS	Small angle X-ray scattering
SBIR	Small Business Innovation Research program
SEM	Scanning electron microscope/microscopy
SEMPA	Scanning electron microscopy with polarization analysis

SET	Single electron transistor
Si	Silicon
SiC	Silicon carbide
SIL	Solid immersion lens microscopy
SIMS	Secondary ion mass spectroscopy
Sm	Samarium
S/N	Signal-to-noise (ratio)
SNR	Same as S/N
SOI	Silicon-on-insulator
SOMA	Self organized magnetic arrays
SPM	Scanning probe microscopy/microscope
SQUID	Superconducting quantum interference device
SRM	Standard Reference Material
STEM	Scanning transmission electron microscopy
STM	Scanning tunneling microscope
SUL	Soft magnetic underlayer
SWNT	Single-walled carbon nanotubes

T

T	Temperature
Tbit (Tb)	Terabit = 10^{12} bits
TEM	Transmission electron microscopy/microscope
TESIL	Tip-enhanced solid immersion lens
TENOM	Tip-enhanced nonlinear optical microscopy
TOPO	Trioctyl phosphine oxide solvent
TXRF	Total reflection X-ray fluorescence

U

UHV	Ultra-high vacuum
ULSI	Ultra-large-scale integration
UMBC	U. of Maryland, Baltimore
UV	Ultra violet
USDA	U.S. Department of Agriculture

V

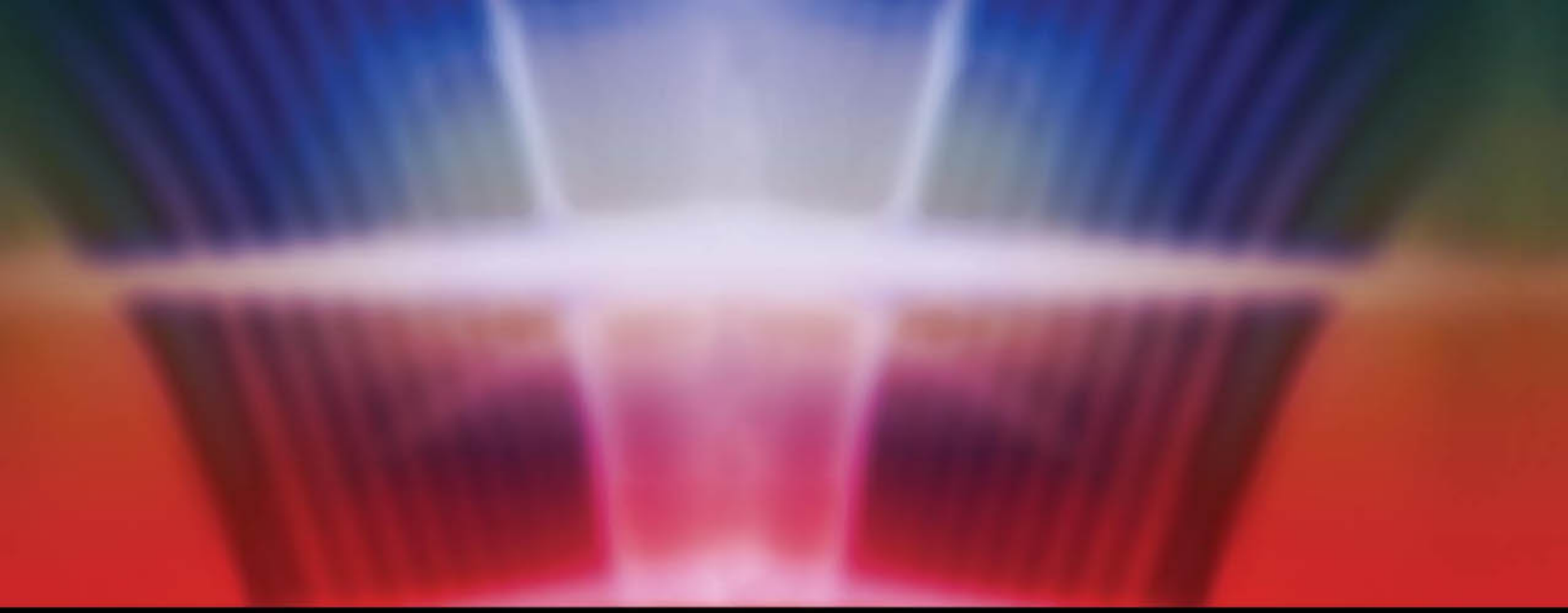
V	Volume
V&V	Verification and validation
VAMAS	Versailles Project on Advanced Materials and Science
VSM	Vibrating sample magnetometer

W

WDS	Wavelength dispersive X-ray spectrophotometer

X

XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
XRF	X-ray fluorescence



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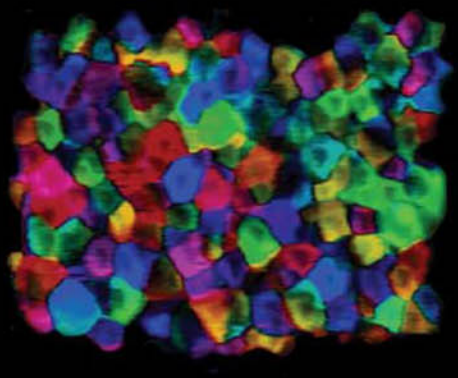
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