Metrology at the Nanoscale: What are the Grand Challenges?

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ABSTRACT

Nanometrology provides the means to measure and characterize nanometer scale process and product performance and covers an expanse of topics including instrumentation, measurement methods (off-line and in-process applications), and standards. To meet the needs of the emerging integrated manufacturing community it is important that research on scale-up of nanotechnology for high rate production, reliability, robustness, yield, efficiency and cost issues for manufactured products and services be pursued. To achieve this, new research directions must include a systems approach that encompasses the characterization of instrumentation, three dimensional metrology, and production hardened metrology. To illustrate the value of metrology and the role of standards to facilitate product realization a number of National Nanotechnology Initiative (NNI) sponsored workshops have been organized. This paper provides an overview of some key findings and recommendations identified at two nanomanufacturing workshops held in 2004 and 2006 that were focused on metrology, instrumentation, and standards.

Keywords: Nanomanufacturing, Integration, Instrumentation, Interoperability, Standards, Nanometrology, Information Technologies, IT, Information Management

1.0 INTRODUCTION

Nanometrology is an integral part of the manufacturing processes for nanomaterials and products that incorporate nanometer scale elements and structures [1]. The success of nanomanufacturing will be dependent on the development of the necessary instrumentation, metrology, and standards that support laboratory research and production organizations. This includes the design and implementation of information technology (IT) systems that enable functionality of a single instrument through the use of algorithms and models, the collective functionality of multiple instruments through integration and interoperability processes, and the development of appropriate information management systems, each of which are critical elements for viable nanomanufacturing. Advanced instrumentation, metrology, and standards will allow measurement and characterization of the physical dimensions, properties, functionality, and purity of the materials, processes, tools, systems, products, and emissions that will constitute nanomanufacturing. There is a need to promote the research, development and application of measurement sciences to enable assessment of the performance of ultra-accurate, off-line, human-in-the-loop instrumentation, and automated production systems for monitoring and control of nanometer scale manufacturing systems. Appropriate attention to these challenges will enable production to be scalable, controllable, predictable, repeatable, safe, and responsive to production environments, requirements, and timeframes. This is the key to meeting the marketplace needs.

2.0 NANOMANUFACTURING

A report of the Interagency Working Group on Manufacturing Research and Development (IWG-MRD) [2] defines nanomanufacturing as "all manufacturing activities that collectively support practical approaches to designing, producing, controlling, modifying, manipulating, and assembling nanoscale elements or features for the purpose of realizing products or systems that exploit properties seen at the nanoscale." Nanomanufacturing is the essential bridge between the discoveries of nanoscience and real-world nanotechnology-enabled products; it is the vehicle by which the Nation and the World will realize the promise of major technological innovation across a spectrum of products that will affect virtually every industrial sector. For nanotechnology products to achieve the broad impacts envisioned, they must be manufactured in market-appropriate quantities by reliable, repeatable, economical, and commercially viable methods. In addition, they must be manufactured so that environmental and human health concerns are met, worker safety issues are appropriately assessed and handled, and liability issues are addressed.

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Nanomanufacturing research and development provides the means to produce reliable tools and processes for precisely manipulating and assembling the basic building blocks of nanotechnology products, cost-effectively producing these products in large quantities, and integrating them into systems spanning nanoscale to large-scale dimensions [2, 3].

Nanomanufacturing includes processes such as:

- the synthesis and processing of nanoelements or nanoscale building blocks such as nanotubes, nanoparticles, nanofibers, and quantum dots;
- nanoelement dispersion in solutions and nanocomposites and atomic-layer deposition for nanoelectronics;
- patterning and templating of polymeric and biomolecular systems;
- directed assembly of 2D and 3D structures and devices;
- positioning, imaging, and measurement at nanoscale resolution; and
- modeling and simulation of material-energy interactions and manufacturing processes at the nano-, micro-, meso-, and macro-scales.

3.0 NIST FOCUS AREA FOR NANOMANUFACTURING

The Manufacturing Engineering Laboratory (MEL) at NIST supports nanomanufacturing [4] by developing a measurement infrastructure that reduces the barriers for technological innovation and successful commercialization of nanoscale products. This is accomplished through fundamental scientific research, theory, and experiments in precision metrology at the nanoscale in the manufacturing sector. The following focus areas are emphasized.

- Reliable, scaled-up, cost effective manufacturing of nanoscale materials, devices and systems
- Robust manufacturing practices coupled with the necessary standards and measurement infrastructure
- Development of the instrumentation, tools and processes needed to bridge the gap between discovery and commercialization.
- Development of a measurement infrastructure and standards for nanomanufacturing to reduce the barriers for technological innovation and successful commercialization of nanoscale products.

The Program is comprised of three themes: Imaging Metrology and Modeling, Nanofabrication, and Control and Assembly. Integration, interoperability, and information management have been defined as cross-cutting technologies.

4.0 WORKSHOP - INSTRUMENTATION AND METROLOGY FOR NANOTECHNOLOGY, JANUARY 2004

A National Nanotechnology Initiative (NNI) Interagency Workshop entitled “Instrumentation and Metrology for Nanotechnology Grand Challenges” was held January 27-29, 2004 at the National Institute of Standards and Technology campus in Gaithersburg, Maryland. The workshop was attended by over 200 nanotechnology experts from industry, academe, Federal laboratories, and private research institutes. The workshop’s objective was to gain input from stakeholders in the field of nanotechnology on the requirements for instrumentation and metrology to support this field and the R&D necessary to develop those capabilities. A new nanotechnology-based industry will require high performance, cost-effective, reliable instrumentation, improved science-based measurement methods (metrology) and globally-accepted standards for measurement and identification of properties and structures at the nanoscale. Accurate measurement of dimensions, characterization of materials, and elucidation of structures at the nanoscale will be critical to the future commercial development of nanoscale materials and devices. As new nanostructures are fabricated, assembled and manufactured into usable products, standardized instrumentation and metrology will be vital for to providing quality control and ensuring reproducible performance.

The workshop results are presented in a report [5] that is available through the NNI (http://www.nano.gov/). Each breakout session included plenary presentations to provide one 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 identifies and highlights research needs in five priority areas for nanoscale instrumentation and metrology.

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 nanoscale identation and nanotribilogy as applied to the mechanics of constrained volume materials.

3. **Nanoelectronics, nanomagnetics, and nanophotonics:** 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:** metrology 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.

The remainder of this section will highlight some of the recommendations identified during the Nanomanufacturing Breakout Session. Below is a short description that was used as the initial starting point for the group discussing the topic "Instrumentation and Metrology for Nanomanufacturing."

Instrumentation and Metrology for Nanomanufacturing: 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 evolution of most of the current metrology technologies such as near-field optics, scanning microscopy, spectroscopy, and interferometry, although the form factor will likely be different to accommodate specific manufacturing requirements. Equally significant will be the development of new automated tools designed specifically for mass production applications. Industry will look for metrology tools that do not require ultra high vacuum (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 through-put yet essential for transitioning the nanoproduct from prototype to production status. Measurement technologies must provide production personnel with the necessary information to maintain control of the manufacturing process to ensure products produced will conform to engineering specifications. This control information must be provided in the timeframes required by production and in formats suitable for production personnel.

Defining the needs for this topic area was challenging as nanomanufacturing covers a diverse field, thus requiring a diverse set of metrology tools and infrastructure suitable for both low- and high-volume markets. It was concluded that first generation tools would likely include variations of the current technologies (see Table 1). Concurrently, new classes of tools would be developed specifically for mass production that support rapid set-up (calibration), reconfiguration for other applications, and ease-of-use by manufacturing personnel.

Table 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 beam (FIB)	Measurement approaches using photons of various energies ranging from optical to x- and gamma ray sources ▪ Scanning near-field optical microscope ▪ Scanning interferometric aperture-less 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 ▪ Auto-alignment and registration Nanometer scale positioning, manipulation, and scanning Actuators

During the course of the workshop there were discussions both on the development of new metrology instrumentation and infrastructure for laboratory research as well as for nanomanufacturing production operations. Laboratory research demands state-of-the-art instruments with extreme precision and accuracy yet typically do not address production attributes such as speed to measure and ease of use. Metrology and instrumentation for the plant floor must be designed to exist in a production environment and be capable of measuring product and process attributes required for conformance testing. The tools must also provide for rapid set-up (i.e., calibration), support reconfiguration for other uses, and support use by manufacturing personnel. There are also demanding requirements regarding precision and through-put that are necessary for transitioning the nanoproduct from prototype to production status**.** Based on these findings the workshop discussion group developed a vision statement for instrumentation and metrology for nanomanufacturing.

Vision for Nanomanufacturing Instrumentation and 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, and 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.

Table 2 Grand Challenges for Instrumentation, Metrology, and Standards

Metrology	Applications and associated challenges/barriers
Real-time decision support for manufacturing	*APPLICATIONS: Production lines with massive sensor arrays, real time response essential for operation, and need to transmit massive amounts of data to and from sensors/tools/instruments in a suitable format at low cost* ▪ Manufacturing / measurement systems that support interoperability and integration allowing for the collection, transfer, and analysis of data between and among those systems that will be integral to the manufacture of products at the nanoscale ▪ System-level models that integrate nanomanufacturing components, sensors, actuators, and control units ▪ Systems/devices that support real-time data collection allowing for fast analysis and transformation into information and knowledge ▪ Properties and theories describing macroscale effects that must be redefined so they apply at nanoscale ▪ Multiscale computational methods that combine dynamics (molecular, coarse-grained mesoscopic and stochastic) and continuous theories ▪ Integrating and analyzing large complex arrays of devices/components ▪ Computationally intensive capabilities (high-speed and parallel processing, at source calculations, sophisticated control algorithms)
"Full Device" inspection with nanometer resolution	*APPLICATIONS: Defect identification, critical dimension measurement, registration* ▪ Single set of requirements for substrate stages ▪ Three dimensional metrology at low cost ▪ Image materials at atomic resolution, analyze material composition with a sensitivity of a few atoms, determine their electrical and bonding properties on a scale smaller than one nm, and complete within a timeframe suitable for mfg. ▪ Inspection tools with 1 nanometer resolution and an overall precision and accuracy of ~0.1 nanometers (corresponds to stage error on order of 10 pm). Tools need to scan across 300-500mm lengths in times consistent with production requirements at suitable costs to meet marketplace needs. ▪ High accuracy stages that minimize susceptibility of stage to environmental disturbances (atmospheric temperature, humidity and pressure fluctuation; vibration, acoustic, and electromagnetic disturbances
Metrology for liquid-phase manufacturing of nanomaterials and devices	*APPLICATIONS: Metrology systems that support measurement of nanoelements such as CNT's and quantum dot properties in liquid phase including soft disordered and interfacial systems and dense collections of nanoelements.* ▪ Visualization of nanoparticles in biological systems with resolutions below and above the 100 to 200 nm scales in the liquid phase with non-destructive methods. ▪ Measure critical dimensions (length, diameter), functionality, surface properties, chirality, of nanoelements and quantify dispersion/aggregation. ▪ Systems measurements vs. nanoelement (interfacial effects, influence on contacts on measurements) ▪ Scalability of manufacturing measurement processes to commercial production rates (e.g., loss of quality, environmentally unfriendly processes) ▪ Measure and deconvolute signal for highly polydisperse, complex systems ▪ Measure static and dynamic properties under different conditions (e.g., process flow – SWCNTs in liquids) and scattering in single walled carbon nanotubes (SCNTs) ▪ Measure and monitor liquid-phase growth of nano-elements -- growth rates and critical dimensions such as real-time monitoring of size and surface structure of growing quantum dots in colloidal dispersion for online control of process parameters. ▪ Measure and monitor liquid-phase assembly of nano-elements of one or more types into larger, ordered nanostructures (e.g. CNTs mesophases) ▪ Model and simulate dispersion and self-assembly phases. Create and correlate models to support process control

The breakout sessions identified three challenge areas that would positively impact the successful deployment of nanomanufacturing technologies if certain barriers were overcome. Table 2 and the three topic areas below provide a summary of the research challenges.

1. *Real-time decision support for nanomanufacturing (models, theory and experiment):*

As 3D nanomanufacturing systems are developed and built, there will be a need to 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)*

Full device inspection requires the development of precision tools for rapid accurate positioning and measurement of planar patterns and 3D structures for the purpose of process development and control. Enabling technologies include rapid imaging and characterization of 3D structure and topography on the surface. For example, the electronics sector has need for stages that support rapid patterning and inspection of sub-10nm lithography features and placement of ~1nm single wall carbon nanotube devices. To validate the tool performance will require standardized parameters and associated testing procedures to specify, describe, and verify the performance of each tool and process used for nanoscale measurement and manufacturing

3. *Metrology for liquid-phase manufacturing of nanomaterials*

New nanometrology tools for in-process control and monitoring of liquid-phase physical and chemical properties for nanoelements and nanocomposites is lacking. Challenges include characterization of process equipment, scalability, quality control, reproducibility, and real-time monitoring of the process. Information technology (IT) will play an integral part for liquid phase manufacturing processes and modeling and simulation (M&S) is required to support novel control strategies. To achieve this capability will require that M&S understand all phases of processing that are important for fluid-to-solid phase processing of nanomaterials. Another key area is the development of techniques for characterizing dense collections of nano-elements under manufacturing conditions.

Reliable, reproducible nanomanufacturing supported throughout the entire production process by rapid, accurate metrology and instrumentation is the key to achieving the economic potential of nanotechnology. The outcome of the nanomanufacturing session was a report [5] that listed technologies that can meet the metrology needs for production, along with a high level roadmap that captured the progression of research milestones required to transition these technologies to production.

5.0 WORKSHOP - INSTRUMENTATION, METROLOGY, AND STANDARDS FOR NANOMANUFACTURING, OCTOBER 2006

The Interagency Working Group on Manufacturing Research and Development (IWG-MRD) sponsored workshop entitled "Workshop on Instrumentation, Metrology, and Standards for Nanomanufacturing" was held on October 17-19, 2006 in Gaithersburg, MD [6]. The workshop's objective was to orchestrate a coordinated effort to support and develop instrumentation, metrology, and standards that enable design and manufacture of materials through improved understanding of mechanisms, processes, and structure-property relationships at the nanometer and molecular scale. This workshop was attended by over 200 experts in nanomanufacturing from industry, academia, and Federal agencies.

The plenary session included invited presentations from top industry experts representing firms manufacturing nanotechnology-related products and support equipment. There were talks on previous National Nanotechnology Initiative (NNI) Grand Challenge workshops that served as a historical review and to set the proper level of discussions for the workshop. The report on the Instrumentation and Metrology for Nanotechnology workshop held on 27–29 January 2004 [section 4.0] at NIST was of particular importance as it was structured around technology challenges and research needs associated with nanomanufacturing of devices and products. To effectively organize the discussions during the workshop and to structure the results, four critical areas were defined; chemicals, electronics, pharmaceuticals, and composite materials.

1. <u>Chemicals:</u> Chemistry serves as a foundation for many aspects of nanomanufacturing, from life science to materials to electronics products. Metrology is an important contributor for understanding and controlling chemistry which is the basis for bonding of different classes of systems (ionic, covalent, metallic, for organic, inorganic, ceramics, semiconductors, metals, etc), non-bonded interactions, kinetics and dynamics, composition/morphology, self-assembly, and chemical reactivity. The breakout sessions were focused on substrate-supported nanostructures and dispersed systems. The topics covered were synthesis, scale-up, functionalization, and assembly of carbon nanostructures, bio-nanomaterials, nanowires, and nanoparticles.

2. <u>Nano-electronics, -magnetics, and –photonics:</u> The semiconductor industry has been pushing device feature sizes down to nanoscale dimensions and continued market pressure keeps this industry searching for new technologies to decrease device size and increase functionality. This includes traditional approaches with new materials and processes as well as disruptive approaches that can offer great benefits yet would require changes to today's foundry processes. In nanomagnetics, physical measurement standards are needed to enable fabrication of magnetic structures with dimensions of 1–10 nm, to measure their chemistry and structure, to measure the magnetization vector of each atom and nanoparticle in these structures and their interactions, and to image magnetic domain structure at 1 nm resolution at high speed. In nanophotonics the needs include (1) instrumentation and metrology to support the development and seamless integration of nanophotonic and nanoplasmonic materials and components into photonic, electronic, and hybrid circuits, and (2)

advanced nanophotonic-based characterization tools, such as near-field scanning optical microscopy (NSOM), for nanoscale 3D imaging and spectroscopic chemical analysis. These include correlation of physical characterization with the electrical properties of devices; fast, non-invasive subsurface/volumetric measurement capability; and 3D-resolved, nondestructive evaluation (NDE) of chemical, physical, electrical, optical, and other properties with nanometer-resolution capability.

3. <u>Pharmaceuticals (Pharma)/Biomedical:</u> The manufacture of new medical treatments and the production of new pharmaceuticals whose functionality is enabled through nanotechnology is a promising market that raises public health and safety concerns and is the subject of strict regulatory restrictions. The pharmaceutical industry has a reputation for the production of high-quality, safe products yet the risks are high for nanopharmaceuticals. Nano pharmaceuticals can be broadly classified into two groups; Nanoengineered Drugs and Nanocarriers.

4. <u>Composites:</u> Nanoparticles and polymer nanocomposite technology comprise a broad and interdisciplinary research and development activity that has been growing rapidly worldwide since 2004. The hoped-for revolution in the nanocomposite industry depends on a variety of state-of-the-art instruments, facilities, and standards for the manufacturing, testing, and characterization of these materials. The requisite instruments include those that can provide detailed information on multiple properties of nanomaterials and nanocomposites simultaneously (magnetic, mechanical, electrical, optical, etc.) and at the nanoscale. Two requirements are the need for nondestructive techniques to probe the buried interfaces, and the need for a set of tools to monitor *in situ* the fabrication and properties of nanocomposites.

Process and Quality Control Needs	**Comprehensive Metrology Needs**
• High-throughput characterization methods, either in-process or off-line • Techniques for materials separation, purification, and classification • Tools to verify purity, quality, and consistency • Tools for testing performance in real-world environments • Instrumentation to support 3D manipulation, assembly, processing, and integration at the nanoscale	• Instruments capable of 3D chemical characterization: elemental & molecular • Platforms for simultaneously assessing multiple properties on a single sample • Techniques that can span multiple length and time scales simultaneously • Tools that can assess chemical reactivity at surfaces and interfaces • Methods for measuring and visualizing chemical dynamics: time-resolved chemistry

As in other areas of nanotechnology, research and development of standards and reference materials is essential for making nanocomposite manufacturing technology move forward. These standards are needed for the consistent manufacturing and reliable characterization and testing of nanocomposites. The ultimate goal is to link key, easily controlled process parameters to nanoscale morphologies/features that define a material's performance. In this way, the understanding of nanoscale features through highly advanced metrology enables the creation of robust process-control parameters that ensure repeatable manufacturing processes that are cost-effective. Federal agencies participating in the National Nanotechnology Initiative (NNI) have established over 30 centers or user facilities and related infrastructure; five of those are devoted to nanomaterials—however, none of those deals exclusively with metrologies for polymer nanocomposites, their processing, or their properties. To rapidly advance nanocomposite technology, it is essential for that kind of infrastructure to be established in new facilities or to be built within the existing facilities.

Workshop discussions yielded recommendations for future research to enable the manufacture of real-world nanotechnology products, and they will help guide the IWG in its efforts to assist U.S. manufacturers in leveraging these efforts into a competitive technological advantage. In addition, the report addresses a cross-cutting environmental, health, and safety (EHS) component that was identified and discussed at the workshop.

TECHNOLOGY ROADMAP

Prior roadmap development efforts provided the workshop participants some insight into the difficult task facing the development of an Instrumentation and Metrology for Nanomanufacturing (NM) Roadmap. The semiconductor industry has realized tremendous benefits from the establishment and continuous updates of its International Technology Roadmap for Semiconductors (ITRS) that provides guidance for its sector's future needs. Each of the four disciplines covered in this report would include requirements on cross-cutting needs like reference materials and instrumentation for nanomanufacturing while also covering unique discipline focus areas such as nanoscale catalysts (chemical discipline). Like the ITRS, the NM roadmap would also provide guidance to instrument manufacturers on reasonable lead times for providing needed tools. It is recognized that the nascent nanomanufacturing industry is vastly different than the semiconductor industry. The nanomanufacturing industry has no consensus on what are the key products, applications, appropriate (maximum impact) initial standards, and common instrumentation and metrology needs. It was recognized by the workshop participants that the NM roadmap envisioned will go beyond the topics discussed in this workshop.

1. The ultimate goal of the roadmap would be to achieve “nanomaterials by design.” The strategy to achieve this goal would include the development of (1) fundamental understanding and synthesis, (2) manufacturing and processing, (3) characterization tools, (4) modeling and simulation, (5) environment, health, and safety protection, (6) standards and informatics, (7) knowledge and technology transfer, (8) education and training, and (9) infrastructure and enabling resources

STANDARD REFERENCE MATERIALS

Standard Reference Materials are essential for comparing measurements taken by different instruments and laboratories. Researchers, material and chemical manufacturers, and product developers must be able to confidently compare and reproduce the properties of new materials and associated processes. Production runs face challenges in controlling process variability which illustrates the need for standard reference materials that ensure existing and new measurement methods are properly calibrated. The use of standard reference materials allow for a deeper understanding of background noise, defects, and other external impacts and provides insight into why an ideal model differs from real-world circumstances. As nanotechnology matures it is expected that an entire library of high-quality reference materials will be developed to address a full range of properties of interest for nanomaterials and the full range of material classes. Some attributes include materials that (a) are widely available and have known properties, (b) have the largest current and potential economic impact, (c) would provide the most meaningful data, (d) could be readily modeled, (e) would meet the requirements of potential users, and (f) are consistent with environmental, health, and safety (EHS) issues.

1. Establish and populate a database of physical property data for nanomaterials (e.g., NIST Chemical WebBook (http://webbook.nist.gov/)) Coordinate development of prioritized list of standard reference materials and plan systematic studies to measure the physical properties of nanomaterials and build a property database.
   - Develop network of databases (distributed repositories) of biological and physical characterization data for nanoparticles that correlate size, shape, concentration, and other physical parameters to its properties, functions, and toxicity
   - Library of high-quality reference materials to address a full range of properties of interest for nanomaterials and the full range of material classes.
   - Identify nanostructured materials that will facilitate cross-laboratory comparisons and trade, and serve as the basis of testing for environmental, health and safety effects
2. Identify generalized reference systems (e.g., the next “fruit fly” -- gold nanoparticle reference material) that serve as basis for metrology for many applications) (e.g., standard reference nanocomposites)
3. Design discovery platform reference systems that provide an advanced starting point for support in validating concepts, fabricating prototypes or serving as test vehicles. (e.g., a wafer with number of structures and functions on its surface on which people deposit their own electronic layers, interconnects and devices or a collection of reference structures for metrology).

STANDARDS METHODS, PROCEDURES, AND DATA FORMATS

Standard methods, procedures, and data formats provide for the consistency and structure that is necessary for the exchange of data in a meaningful and effective way. By taking this approach a team has the tools to better coordinate research activities and understand the results of individual and shared research results.

1. Define standard operating procedures (SOPs) for the synthesis of nanomaterials and sample preparation procedures for measuring, handling and storing these materials.
   - Standard processes for creating uniform nanoparticles for test and characterization.
   - Development of standard test protocols for toxicity coordinated among community stakeholders.
   - Investigate applicability/feasibility of microfluidics techniques for manufacturing processes and in addressing EHS concerns.
   - Solution-based metrologies to disperse nanoparticles so that advanced characterization studies can be carried out.
2. Examine and develop standard labeling practices and Material Safety Data Sheets (MSDS) requirements for nanomaterials.
3. Develop standard formats (e.g., meta-data) for the capture, storage, retrieval, and interpretation of raw data so that data quality and integrity can be evaluated
   - Develop ontology/data dictionaries (e.g., similar to program in place already in the cancer research community); XML-based applications and object-models.
   - Standard methodology to define size, shape, concentration, and other physical parameters of nanoparticles (includes platelets, nanotubes, and nanofibers)

MODELING

Effective implementation of modeling and simulation (M&S) practices should include multidiscipline and multiscale application. Modeling and simulation is an enabling tool for material, product, and process discovery, product and process design, and process control. The development of predictive models to discover new materials based on physical property data and reference materials is essential. Successful M&S requires the user to coordinate integration efforts and access to high end computing. For many applications in nanomanufacturing, complex modeling and high-performance computing are critical to nanoscale metrology as the behavior of discrete atoms becomes important at nanoscale dimensions. Further, interactions between measuring devices and samples make quantitative measurements of material properties extremely challenging. The interpretation of nanoscale imaging and the modeling of nanoscale devices and systems are severely limited due to limitations in computational speed.

1. Support development of predictive multiscale models to discover new materials based on physical property data and reference materials, and for process development, control, and predicting product performance and life cycle.
   - Computational models for nanocomposites that provide predictive capability for correlating electrical, thermal, mechanical, and acoustic properties to the synthesis and manufacturing-processes and quality-control (QC) metrics.
2. Ability to observe the structure of the nanoparticles in its three-dimensional matrix would enable better models of the transport to be developed.
   - Three-dimensional visualization of structure at the nanoscale. Transport phenomena, such as electrical and thermal conduction, stress transfer and viscous dissipation are governed by particle-particle interactions as mediated by the matrix.
   - Understand and adapt to scanning probe’s inherent non-linearity and drift. Uncertainties of the tip structure lead to uncertainties in measurement restricting imaging to near atomically flat surfaces.

NEW INSTRUMENTS AND TOOLS

New measurement tools are needed that range from the highly sophisticated to the relatively simple. This is a key enabler for understanding the link between process, structure, and functionality that is essential for the creation of new nanomaterials and the development and optimization of new synthesis methods. Sophisticated instrumentation will enable the fundamental understanding of chemical and material behavior at the nanoscale and how materials form and react. Innovative yet less complex instrumentation is needed for use in manufacturing environments for process control, quality control, material classification, and reliability testing. This instrumentation must support rapid identification of material purity and homogeneity, while remaining relatively easy to use.

1. Develop advanced, off-line material characterization tools that emphasize ways to increase the functionality of existing instrumentation (AFM, SEM, TEM, etc.) making the tools multifunctional and capable of working with combinatorial methods. Some measurement functionalities identified include:
   - characterize the adhesion between the matrix and the nanoparticle, measurement methods for stress transfer, and kinetics
   - characterize dynamic changes in local dipole alignment, spin orientation, stress, plasmon properties could enable the understanding of the coupling of the phenomena
   - characterize the control of self assembled features and their alignment to previously fabricated structures
   - high resolution photoelectron emission microscopy, x-ray phase sensitive reflection, near field microscopy with SiC superlenses, AFM/capacitance and impedance spectroscopy
   - scanning probes that support atomic control of the tip and shape
   - novel probes to detect spin, plasmons, molecular state, etc. with atomic resolution at surfaces and interfaces
2. Develop advanced, off-line measurement tools that supports
   - probe buried interfaces at the nanoscale through non-destructive techniques
   - measurements that can span multiple length and time scales simultaneously.
   - probe to understand chemical reactivity at the interfaces.
   - measuring and visualizing chemical dynamics; time-resolved chemistry.
3. Develop advanced, in-line material measurement tools that supports
   - real-time monitoring of manufacturing process for real-time process development
   - high-through put of materials during production with measurement accuracies that support good decisions regarding process performance and product quality
   - determination of complex relationship between processing, rheology and final properties (such as conductivity) to design cost-effective strategies to manufacture materials with existing equipment in which sizable capital expenditures have been made.

- implemention of complex sampling strategies and statistical methods for new processes and capability of automated real-time transition from identified areas of interest at wafer level scales (100-300mm) to precise measurements at device level scales (<100nm).
- real-time monitoring of self-assembly processes, including 3D methods capable of probing through layer depths (e.g., primary inspection techniques such as ellipsometry or Raman, higher resolution, secondary techniques, registry tools to integrate bottom-up with top-down self-assembly processes.
- measurement of nanoparticle dispersion during synthesis, throughout the manufacturing process, and into the final manufactured part.
- collection of experimental data of nanoparticle formation to support development of computational models for formation process.
- scale-up, and control; for quality control; and for EHS monitoring and control.
- development of tools that can monitor, *in situ*, the fabrication and properties of nanocomposites.
- design of miniaturized instruments for process monitoring and control.
- new approaches to achieving real time protein binding and control instrumentation.
- methods to image living biological structures without damaging or killing them in an environment compatible with living biological samples.

CHARACTERIZATION

Characterization is founded on the development of basic measurement metrology at the nanoscale for the determination of bulk and surface material properties and for process monitoring. Measurement methods are being developed for use in conjunction with new instrumentation and calibration artifacts. The scope of the discussion encompasses metals, ceramics, and polymers in various forms — particles, thin films, nanotubes, and self-assembled structures — and also includes studies of nanocomposites and liquid-state properties for microfluidics-based fabrication and measurement techniques. Physical properties such as mechanical strength, elastic moduli, friction, stiction, adhesion, and fatigue strength are measured, as well as the size of nanoparticles and the structure and dispersion behavior of nanoparticulate systems. Other properties such as electrical conductivity, thermal conductivity, magnetic properties, electronic properties, and optical properties are also examined. While the discussions focused on developing measurement techniques at the nanoscale, proper data interpretation requires fundamental studies in nanomechanics, scaling laws, and imaging techniques.

1. Develop standard characterization methods unique to each class of material for characterization: nanotubes, platelets, fibers and nanoparticles.
2. Develop reliable parameters for measurement of dimensions, electronic and optical properties and performance.
3. Develop advanced metrologies that will support rapid measurement of large numbers of particles (100s or 1000s) so that accurate statistics of particle distributions can be obtained.
4. Develop solution-based metrologies to disperse nanoparticles that allow advanced characterization studies to be carried out.
5. Develop characterization techniques for instruments used to measure the processes and products.
6. Framework to characterize changes in the matrix due to addition of nanoparticles (interphase) such as the creation of new crystal morphologies, distortions in polymer chain conformation, or mobility.
7. Framework to characterize the changes to composite properties upon addition of nanoparticles (rheology, strength, toughness, conductivity, permeability) with the size, interfacial chemistry and intrinsic properties of the nanoparticles.
8. Framework to characterize how the changes in matrix structure and composite properties affect the performance of the composite in applications (product stability, processability, spreadability, vapor-barrier properties, heat deflection, etc.).
9. Framework to characterize nanoparticles that are introduced directly into a fibrous preform independently from the matrix via processes such as chemical vapor deposition and the like and linking these characteristics to resultant composite properties.
10. Framework to characterize nanostructure and multiple properties simultaneously at the nanometer scale. Research should be pursued in sources, the physics of the source-probe-sample interaction, novel detectors and especially models to enable decoupling the probe sample interactions and delineate the structure and properties… [including] novel probe techniques that can monitor the dynamic response of multiple properties to applied stimuli.

ENVIRONMENTAL, HEALTH, AND SAFETY

Shared industrial metrology needs exist across aerospace, automotive, chemical, forest products, pharmaceutical, and semiconductor industries. Advanced measurement science will be necessary, for example, to detect trace levels of exposure to nanomaterials resulting from medical, occupational, environmental, or accidental release. Hence, it is essential to develop the instrumentation and metrology to accurately follow the environmental fate of nanomaterials, develop safe nanoscale sample handling methods, and accurately measure the effects throughout the entire product lifecycle. Manufacturing plant air needs to be monitored for particle concentration and size characteristics to determine if nanoparticles are released during manufacture. If nanoparticles are found to be present in plant air, companies must evaluate the plant's conventional air handling and conditioning system and consider whether it is effective against nanoparticles and offers protection to the workplace environment.

1. Worker monitoring of and exposure to nanomaterials
2. Customer exposure to nanomaterials
3. Waste management
4. Life cycle planning and testing

INFRASTRUCTURE

A key challenge that researchers face is securing the resources necessary to achieve quality, consistency, and cost targets when scaling up material production from laboratory-scale to production-scale operations. This is further exacerbated by the high cost of equipment and the need for dedicated operators for running the instrument or equipment. Centralized facilities with experts available for enabling the development and use of exotic and emerging techniques are essential for companies and many universities that can not afford the equipment to support their research.

1. NNI or other agency centers and user facilities that deal exclusively with metrologies for polymer nanocomposites, their processing, and their properties.
2. Protection of intellectual property

6.0 SUMMARY

Significant progress has been made in developing advanced instrumentation and metrology that clearly demonstrates that monitoring and control of certain nanomanufacturing processes at required quality levels is achievable. Leveraging these advances with statistical methods and novel sampling strategies will further enhance this capability. Other advances in measurement technologies that support the characterization of nanoscale material properties will increase a manufacturer's capability to maintain process knowledge within its own enterprise and to work effectively with their extended supplier base. With the development of new instrumentation and metrology, combined with appropriate best practices and standards, new processes will support manufacturers in the production of high quality, high value products which meet market expectations. As noted by both workshops, IT plays a key role in creating this cohesive toolbox of technologies that will support metrology and instrumentation advances necessary for responsive and valued knowledge creation and information exchange. Recent advances in integration, interoperability, and information management [7] technologies, and associated fields such as math and computational science and digital libraries, will provide an essential ingredient for new measurement systems and their ability to manage the massive amount of information that will be generated. A dramatic increase in worldwide investments over the past few years in nanoscience research has resulted in new scientific breakthroughs that will soon look to nanomanufacturing for the creation of products.

A common theme of each sector was the need to develop and provide a practical and essential array of instrumentation, metrology, and standards to understand and manage environmental, health, and safety issues (EHS). Each sector identified broad needs for fundamental nomenclature, metrology, instruments, and standards including nanomaterial and nanoparticle standard reference materials of different media, size, chemical composition, surface chemical functionality, shape, and charge that will enable traceability, calibration, and validation of measurement tools used in nanomanufacturing process monitoring and EHS applications. There are a number of reports which state the importance of continued and increasing research regarding EHS of nanomaterials. Three key reports are the Chemical Industry Vision2020 Technology Partnership report (2003), the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council's Committee on Technology document that identifies EHS research and information needs (NSET 2003), and the NIOSH Web document "Approaches to Safe Nanotechnology: An Information Exchange with NIOSH" (NIOSH 2006). These documents are intended to assist industry in ensuring that proper precautions and measures are followed to ensure that no worker suffers material impairment of safety or health as nanotechnology develops.

7.0 CONCLUSION

To meet the identified metrology, instrumentation, and standards challenges, it will be necessary to develop an effective alliance of government members for funding and research, academic participants to cultivate innovation in science and engineering, and industrial partners to rapid assimilate and apply new knowledge into the design and production of affordable, competitive new products.

- Reliable, reproducible nanomanufacturing supported by rapid, accurate metrology and instrumentation is the key to achieving the economic potential of nanotechnology

- Existing metrology tools need to be dramatically improved, and innovative tools based on entirely new ideas will need to be developed.

- Production metrology must 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.

Advanced nanomanufacturing is the key to the strength and future growth of the U.S. manufacturing sector and a strong measurements and standards infrastructure is vital for its success. The degree to which the research communities are able to pursue the recommendations presented in these two workshop reports will dictate the resulting outcomes for improved U.S. product quality and manufacturing process performance, reduced production costs and time-to-market, and increased competitiveness in international markets. NIST is responsible to U. S. manufacturing for providing traceability to the national unit of length, by developing measurement capabilities and calibration standards and is working to provide these needed components. It is hoped that this paper and the workshop reports will set the stage for the development of innovative solutions and promising results for the nanomanufacturing and nanometrology communities.

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