MEASUREMENT SCIENCE AND TECHNOLOGY FOR CERAMICS INNOVATIONS

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I. INTRODUCTION

Innovations in ceramic technologies are often driven by the discovery and introduction of a material with novel or improved behavior that enables realization of a superior component, device or system. Over the past few decades, advanced ceramic materials have spurred growth across all technology sectors, for example, gate dielectrics for semiconductor electronics, high frequency dielectrics for microwave communication, and semiconducting light emitting diodes for traffic signals and lighted signs. Looking to the future, ceramic materials will play a vital enabling role in emerging applications such as semiconductor quantum dots for drug delivery, multiferroics for multifunctional sensing devices, and thermoelectrics for vehicular waste heat-recovery. Performance of the components, devices and systems incorporating these and other novel ceramic materials will depend on the behavior of the materials, which, in turn, is determined by the relationships between the structure and properties of the materials. More powerful and rapid standardized measurements of material structure and properties are needed to guide and accelerate materials and process selection and optimization for advanced applications. Nanotechnology is becoming increasingly pervasive, and many existing measurement methods are not capable of probing nanoscale dimensions and entities. Meeting these measurement needs will require the development of measurement science—the underlying physical principles forming the basis for the measurement—as well as measurement technology—the instrumentation, methods, models, and standards enabling an accurate and precise measurement. This paper provides specific examples of how measurement science and technology can enable the development and incorporation of ceramic materials in a wide spectrum of applications.

II. IDENTIFYING NEEDS FOR MEASUREMENT SCIENCE AND TECHNOLOGY

There are a number of effective ways to identify opportunities for measurement science and technology (measurement needs) to overcome barriers to technology innovation. Consensus measurement needs for a specific technology area are commonly addressed by comprehensive industry roadmaps such as the International Technology Roadmap for Semiconductors[1] and the Advanced Ceramics Technology Roadmap[2]. Focused workshops such as the Ceramic Coatings Metrology Workshop[3] and the Nanotechnology and the Environment: Applications and Implications STAR Progress Review Workshop[4] are another means of focusing attention on critical measurement problems.

Many public and private organizations across the U.S. engage in the development of measurement science and technology. In an effort to bring together these disjointed activities and identify the Nation's most critical measurement needs across all technology sectors, the National Institute of Standards and Technology (NIST) has undertaken an ambitious effort to assemble these activities into the United States Measurement System or USMS[5]. The first stage of the USMS exercise involved the initial collection and assessment of two types of measurement needs: (1) individual needs developed by single companies or consortia, teams at USMS-sponsored focused technology workshops, and individual NIST staff members; and (2) roadmap needs taken from over 160 published reports. The over 300 individual measurement needs and 400 roadmap measurement needs included in the exercise are only a small sampling of the needs limiting technology innovation. Of this sample group, the majority of the roadmap measurement needs focused on manufacturing, while most of the individual measurement needs were in the applied research and development stage of the innovation cycle. In Fall 2006, NIST will release an assessment report[6], including: (1) examples of specific industry measurement needs identified as posing technical barriers to innovation; (2) an analysis of the trends and commonalities among these measurement needs; (3) initial findings on the state of the USMS and identification of any systemic problems; and (4) recommended follow-up actions to fulfill these measurement needs.

III. MEASUREMENT NEEDS FOR CERAMIC TECHNOLOGIES

The measurement needs for ceramic technologies presented in this paper were taken largely from the USMS exercise. Of the more than 300 individual USMS measurement needs, about 40 are focused on ceramic materials, taken here to include oxides and other chalcogenides, nitrides, carbides, elemental and compound semiconductors, and concrete. An additional 32 roadmap measurement needs identified in the USMS exercise involved ceramic materials and were mostly concerned with production and quality control, with many calling out the need for improved sensors. Only individual measurement needs were selected for inclusion in this paper, and most fall within the applied research and development stage rather than the manufacturing stage. In addition, the selected needs focus on the development of measurement science and technology to provide solutions to the measurement problems, not the actual measurements *per se*. It should be emphasized that the measurement needs included in this paper are merely illustrative and by no means represent an exhaustive list of the critical needs for a given application of ceramic materials.

The selected needs were classified by device or system and mapped onto the technology sector framework forming the basis of the sessions in the 1st International Congress on Ceramics (ICC). Eight categories of devices and systems were chosen and ordered in increasing length scale from smallest (nanoscale) to largest (macroscopic): Nanoparticles and Nanocomposites, Integrated Circuits, Magnetic Storage, Lasers and Light Emitting Diodes, Microelectromechanical Systems (MEMS), Energy Generation, Cutting Tools, and Civil Infrastructure. Specific applications of these eight devices or systems in the ICC technology areas—Electronics, Energy, Environment, Glass and Optical, Consumer Products, Biology and Medicine, and Transportation—are presented in Table 1, along with the primary materials for each device or system. Construction was added to the seven ICC Technology areas to capture measurement needs in concrete and insulators, two pervasive ceramic materials of economic importance.

In the following sections, each device or system and its application to ceramic technologies, particularly in emerging areas, is described, along with the primary measurement needs limiting innovation and development. For clarity, the measurement needs and potential solutions to these needs are summarized in tables.

Nanoparticles and Nanocomposites – Table II

Nanoparticle manipulation and detection are being pursued as the basis for next-generation developments in a wide range of applications, including high-density electronics, sensors for biochemical applications, actuators in nanomechanical devices, and biomedical

Table I. Application of Devices, Systems, and Materials in Ceramic Technologies

Device or System	**Material**	**Electronics**	**Energy**	**Environment**	**Glass and Optical**	**Consumer Products**	**Biology and Medicine**	**Transportation**	**Construc-tion**
Nanoparticles Nanocom-posites	Colloidal Si, SnO_2, TiO_2, ZrO_2, SiO_2, SiN, CdSe	CMP[a], field effect transisters	Fuel cell membranes, catalysts, batteries, fuel additives	Sensors for biotoxins, filtration systems, chemical neutralizers	Optical fiber cladding	Paints, sunblocks, clean surfaces, cosmetics, clothing, packaging	Drug delivery, image enhancers, disease treatment	Catalytic converters, wear-resistant coatings, high strength/weight materials	Fire-resistant and high strength / weight materials
Integrated circuits	Si, SiO_x, SiN_x, nanoporous Si-C-O-H, HfO_2, Ba-Sr-Ti-O, ZrO_2, strained Si	Micro-processors, memory				cell phones, portable audio and video equipment, appliances			
Magnetic storage	Al_2O_3, Al_2O_3-TiC, $CaTiO_3$, NiZn ferrites	Data Storage				Set-top boxes, Portable audio and video			
Lasers and LEDS	GaN, AlGaN, GaAs, AlGaAs		Water purification systems	Chemical and biological detectors	Solid state lighting, fiber optic systems	Electronic displays, DVD and CD players	Surgical tools, disease detection and treatment	Vehicle and road signaling, traffic lights, maritime navigation	Building displays
Microelectro-mechanical Systems (MEMS)	c-Si, poly-Si, SiC, SiN_x, $Pb(Zr,Ti)O_3$	Sensors and actuators in hand-held devices	Pressure and chemical sensors	Sensors for bio-toxins	Digital mirrors in optical projectors	Digital mirrors in large screen HDTV's	Bio-MEMS: for surgery, drug release	Sensors and accelerometers in cars	
Energy generation	BiTe, $CeFe_3CoSb_{12}$[b], LSM[c], doped CeO, YSZ[c], LCSF[e] $YBa_2Cu_3O_7$	Device cooling	Engine waste heat-recovery, power systems for buildings, electric power lines, motors	Systems to reduce fossil fuel emissions	Heaters in fiber optic switches	Refrigeration	Controlling temperature in bioreactors	Engine waste heat-recovery	
Cutting tools	WC, diamond, cubic BN	Stepper motors	Generators, electric motors			Sports equipment	Surgical tools	Vehicle engine parts	
Civil Infrastructure	Concrete, gypsum, glass fibers		Power plants, dams, solar heating systems	Levees, dams, solar heating systems					Buildings, bridges, pipes, fire protection, insulation

[a]chemical-mechanical planarization; [b]*i.e.*, skutterudite compounds; [c]$La_{1-x}Sr_xMnO_3$; [d]yttrium-stabilized zirconia; [e]La-Co-Sr-Fe-O

diagnostic and delivery mechanisms. In addition, innovative materials that incorporate nanoparticles are rapidly expanding in many sectors of the world economy, including cosmetics, pharmaceuticals, structural composites, and clothing products. Illustrative measurement needs (MNs) and measurement solutions for nanoparticles are summarized in Table II as MN IIa-IIi.

The large surface area to volume ratio of nanoparticles leads to strong surface interactions between the particles and manipulating probes, between the particles and intended substrates, and between the particles themselves. Hence, understanding and control of the adhesion and bonding of surfaces, particles, and nanoelements comprising a nanodevice is required for device fabrication and optimization. Interactions at the nanoscale, however, are highly dependent on the size, composition, and surface layers of the entities involved. Assumptions of structure based on bulk crystalline structures are known to be inaccurate and do not provide appropriate guidance for nanoparticle manipulation. Methods are required to measure the size and size distributions of nanoparticles and to measure their composition with nm resolution. (MN IIa, Table II)

Control of particle concentration and size distribution is critical in advanced semiconductor manufacturing as it strongly affects device yield and performance. Cost-effective measurement of particle sizes below 90 nm is needed to improve device performance of current and future technologies. Current particle detection metrology is based on optical methods and is limited due to the relatively long wavelength of the optical lasers employed, as well as detector designs and beam optics. A rapid particle measurement system for particle sizes less than 90 nm is required. (MN IIb)

Nanoparticle-based vectors for detection and treatment of cancer will result in precise *in situ* imaging and localization of tumors, sensitive *ex situ* detection, and localized application of chemotherapy directly to tumor cells. Such innovations will provide for early disease detection, improved efficacy of anti-cancer drugs, and decreased toxicity of treatments to non-targeted cells. Accurate measurement of particle size is a starting point for selecting nanoparticles for these applications, but, in addition to this need, there is no standardized, analytical cascade for the physical (physico-chemical) characterization of nanomaterial vectors. (MN IIc) This lack of a standardized cascade is caused by a paucity of measurement tools for the critical material parameters that influence nanomaterial transport, toxicology, and effectiveness in biological systems.

New classes of nanoparticle-based magnetic contrast agents are being developed to dramatically extend the modalities of magnetic resonance imaging (MRI). These contrast agents target specific tissues, metabolic pathways, and neural and cardiac functionalities, enabling new types of imaging of molecular and neural activity and better diagnoses for a broad class of medical problems. The technical barriers to the development of better contrast agents are the ability to nano-engineer contrast agents that have high spin relaxivity, are non-toxic, and can be targeted to the desired organ or tissue. (MN IId)

Despite the potential gains from nanoparticle-based products, there is concern by many communities about their toxicity. The unique and diverse physico-chemical properties of nanoscale materials suggest that toxicological properties may differ from materials of the same or similar composition but of larger size. For example, nanoparticles may readily migrate through normal barrier tissues such as skin, enter into the blood stream and from there penetrate internal barrier tissues and enter organs such as the brain. Effective and accepted measurement methods to assess nanoparticle toxicity do not exist. Evidence demonstrating the migration of nanoparticles through barrier tissues in mammals has only recently been shown. New measurement methods are needed for both clinical effects of nanostructure incorporation in tissues and for characterization of nanostructures throughout their life, *i.e.*, before incorporation into tissues as well as following incorporation. (MN IIe) It is expected that once incorporated in living tissues, nanostructures will change. Methods having the capability to determine such changes are also required.

Directed self-assembly processes for the production of novel materials and devices incorporating nanoparticles and other nanoscale entities have been demonstrated at limited scales. However, these processes have not been developed in the context of large-scale manufacturing or production. Measurement methods to verify fidelity and reproducibility of the structures of self-assembled materials and devices in large numbers are required for commercial success. (MN IIf)

Carbon nanotube composites are a new form of material that promise to yield large strength-to-weight ratio components for airplanes, satellites, flywheels for energy storage, and other applications in which high performance justifies the added cost of the raw materials and fabrication. The key to optimum performance in a fiber-matrix composite material is the strength of the interface between the reinforcing fibers and the matrix. Conventional methods developed for fabrication of fiber-matrix composites with larger, weaker fibers have not succeeded with the new ultra-fine, ultra-strong nanotubes. Proper dispersion of the tubes in the matrix is difficult and the tube-matrix interface seems weak. Inability to measure the atom-scale structures in the as-manufactured material, how they deform, and how they de-bond when the material is stressed impedes the ability to design nanotube-based composites with adequately strong interfaces. (MN IIg)

Thermoplastics and composites made from polymers can improve the affordability, strength-to-weight ratio, and durability of manufactured products. Unfortunately, the use of these materials in buildings and vehicles is limited because of their inherent flammability and poor thermal stability. Nanoadditives, including clays, layered double hydroxides, and nanotubes have been shown to reduce costs, increase thermal stability, reduce flammability, and enhance mechanical properties when they are homogeneously dispersed in polymers. The flammability of polymer nanocomposites depends critically on how the nanoadditives are introduced into the polymer matrix. However, there are no quantitative metrics for assessing the degree of nanoscale mixing, dispersion, or interaction attained in the composite and how this affects the properties of interest. (MN IIh) In the absence of the measurement tools and associated metrics, it is impossible to determine the nature and amount of nanoadditive needed to obtain optimal performance.

All automotive coatings are formulated with photostabilizers to protect the coating from weathering. Recently, the coatings industry has formulated materials with metal oxide nanoparticles that act not only as ultraviolet absorbers but also improve the scratch and mar resistance of a coating. Substitution of these metal oxides for their organic photostabilizer equivalents dramatically reduces the cost of coatings. In addition, automotive companies have employed special effects pigments, aluminum flakes, and interference pigments to differentiate their products from those of their competition. However, the automobile industry cannot improve coating products as they cannot measure their properties with appropriate accuracy or precision, and the effect of pigments on the aging of coatings is unknown. Metrologies for measuring the effectiveness of these additives on appearance and weathering are not available so that implementation of this technology has been hampered. (MN IIi)

Table II. Measurement Needs and Solutions for Nanoparticles and Nanocomposites

Subtopic (MN #)	Measurement Need	Measurement Solution
Size and structure (IIa)	Methods to measure nanoparticle size, surface area, shape, composition, surface layer thickness and composition, crystalline perfection, and interatomic spacing.	High resolution x-ray diffraction metrology and standard reference materials certified for crystallite size, amorphous content, and lattice parameter.
Size (IIb)	Particle measurement systems to accurately measure particle sizes below 90 nm, capable of operation in a semiconductor-manufacturing environment.	New laser sources and scattering techniques and validation of new measurement techniques, including those at synchrotron and neutron facilities. Development of standard methods and data for the new instrumentation.
Properties (IIc)	Methods to measure the physical nature of different nanomaterial classes under physiologic conditions. Properties include hydrodynamic size and size distribution, morphology, aggregation, stability, surface charge, zeta potential, chemical composition, and purity.	Extension of methods for physical characterization of nanoparticles to relevant conditions and that relate physical (material) characteristics to biological interactions (*e.g.,* toxicity, reactivity, mobility) and pharmacokinetics. Relevant reference materials.
Properties – MRI contrast agents (IId)	Tools to measure the effects of functionalization of magnetic contrast agents on relaxivity properties.	Metrology to underpin single particle tracking in MRI systems.
Properties – toxicology (IIe)	A set of metrics that accurately characterize the toxicology of nanoscale materials.	New measurement approaches to determine *in vivo* and *in vitro* properties of nano-structured particles and devices.
Nanocomposites – self-assembly (IIf)	Measurement of selectivity, specificity, and registry of nanoscale building blocks in directed self-assembly.	Quartz crystal microbalance measurements combined with evanescent-wave fluorescence correlation spectroscopy and fluorescence-resonance energy transfer to probe assembly over a (2 to 10) nm length scale and a (100 ns to s) time scale. Model test patterns with known selectivity, specificity, and registry of building blocks.
Nanocomposites – carbon nanotubes (IIg)	Measurement of the atomic-scale three-dimensional structure of carbon nanotube-matrix interfaces.	A nano-electro-mechanical probe with capability to gradually erode a material surface and record the location and chemical identity of each atom as it is removed.
Nanocomposites – dispersion (IIh)	Methods to assess the degree of dispersion in polymer matrix nanocomposites.	Combination of three-dimensional measurements, statistical analysis of experimental data, and standard reference materials.
Coatings – mechanical properties (IIi)	Measurement tools for initial and aged coating appearance.	Correlation of coating scratch resistance, interfacial adhesion, and other nanomechanical properties with optical measurements.

Integrated Circuits – Table III

Integrated circuits are ubiquitous in electronics and consumer products and drive much of the growth in these large technology sectors. Ceramics are vital to all current and future integrated circuits, from high dielectric constant (high *k*) thin films in complementary metal oxide semiconductor (CMOS) devices to dielectrics as embedded passive components for high frequency applications to strain-engineered silicon for next-generation CMOS devices. Integrated circuits are multilayer structures of many dissimilar materials fabricated in multiple processing steps, so materials interactions at the nanoscale are a critical factor in device performance. Illustrative measurement needs for integrated circuits were classified into two broad areas—scaling and enhanced performance—and are listed in Table III.

Further scaling (dimensional shrinkage of integrated circuit device elements according to Moore's Law) in Si microelectronics is limited by the ability to: (1) rapidly introduce new materials with improved properties and (2) characterize composition and structure at the nanoscale. Overcoming both of these limitations will require measurement science research and measurement method development. One dire need for new materials is in the gate stack structure: a material with a dielectric constant greater than about 20 is needed to replace the SiO_2 (dielectric constant ~3.9) gate dielectric, and a compatible metal is needed to replace the degenerately doped polycrystalline Si gate electrode. (MN IIIa, Table III) The selection of the optimal gate dielectric-electrode material combination will require nanoscale measurement of interface stability and electronic properties such as work function and interface state density.

New barrier layer materials are required to separate incompatible materials in integrated circuits at increasingly smaller length scales, *e.g.,* inhibiting copper diffusion from interconnect levels to the device gate where the presence of copper destroys transistor operation. Here a coupled measurement method-modeling approach is needed to evaluate mass transport between the dissimilar materials to predict long-term stability. (MN IIIb)

Scaling also presents formidable challenges for accurately measuring structure, composition, and dopant distribution at the nanoscale, particularly for in-line measurements of real-time monitoring and process control. Device manufacturers predict that measurements at true atomic resolution in three dimensions will ultimately be needed. (MN IIIc) The precision and resolution of existing electron microscopy, scanned probe microscopy, and in-line optical probe methods must be improved to realize atomic scale measurements. Accurate measurement of device dimensions will become increasingly difficult as devices shrink in size and will require improvements in high-throughput characterization methods such as optical scatterometry. (MN IIId)

Enhanced performance—operation at greater speeds with increased power and component densities and more efficient energy usage—drives advances in integrated circuits. Incorporating alternative materials with improved properties is one means of enhancing performance. Strained silicon layers are being developed to increase carrier mobility in next-generation sub-60 nm devices, thereby improving performance without scaling. Existing x-ray and optical methods for determining strain have spatial resolution in the hundreds of nanometers, so novel measurement methods with resolution approaching 10 nm are required for commercial success of strained devices. (MN IIIe) Alternative semiconducting materials wherein electron spins replace charges as the basis for switching and transistor functions are also being considered for introduction into integrated circuits by 2020. Measurements are needed to assess the spin injection and transport properties and measure the optical properties of potential spin-polarizing materials. (MN IIIf)

Table III. Measurement Needs and Solutions for Integrated Circuits

Subtopic (MN #)	Measurement Need	Measurement Solution
Scaling – high *k* gate dielectrics (IIIa)	Methods to assess: (i) interfacial stability; and (ii) key electronic properties such as work function and interface state density. Measurements must be made rapidly as there are thousands of possible combinations of gate dielectric and metal electrode materials compositions.	New high-throughput, combinatorial measurement techniques, such as nano-calorimetry, to measure thermal stability, and capacitance-voltage characteristics to derive the metal-semiconductor work function. Techniques would accelerate the optimization of gate dielectric-metal electrode combinations.
Scaling – diffusion barrier materials (IIIb)	Accurate and rapid methods to assess transport at nanometer length scales through all classes of materials.	X-ray diffraction measurements in combination with thermodynamic and x-ray scattering models to interpret results would permit universal, rapid evaluation of mass transport. Combinatorial or high-throughput techniques would accelerate the identification of optimal materials.
Scaling – composition and structure (IIIc)	In-line tools to measure the composition and structure of ultra-thin films and interfaces with the required resolution, precision, and accuracy. Measurement tools to determine 3D dopant concentration, location, and activation at the nanoscale.	Aberration-corrected electron microscopes such as scanning electron microscopes and transmission electron microscopes (TEM), scanning TEM, and scanned probe microscopes. Improved tools for in-line optical measurements from the far IR to VUV, *e.g.,* polarized reflectivity; spectroscopic ellipsometry; second harmonic generation; X-ray reflectivity, small angle scattering, diffraction, and photoelectron spectroscopy; Auger electron spectroscopy; and higher energy resolution backscattering methods.
Scaling – dimensional control (IIId)	Accurate measurement of device dimensions by a high-throughput technique such as optical scatterometry.	Electromagnetic models to extract optical property data from optical scatterometry measurements, providing greater accuracy.
Enhanced performance – strain-engineering (IIIe)	Innovative measurement methods to map strain with a spatial resolution of ~15 nm.	Tip-enhanced Raman spectroscopy and electron-backscatter-diffraction techniques will potentially extend the spatial resolution of strain measurements to 15 nm to 20 nm.
Enhanced performance – semiconductor spin properties (IIIf)	Measurement methods: (i) to assess spin injection and transport, and decoherence of small quantities of electron spins; and (ii) to determine optical properties of spin-polarizing materials.	Scanned probe methods based on time-resolved Faraday rotation to detect single electrons and their spin properties. Methods to measure the optical properties of spin-polarizing materials.

Table III. Measurement Needs and Solutions for Integrated Circuits (cont.)

Subtopic (MN #)	Measurement Need	Measurement Solution
Enhanced performance – embedded passives (IIIg)	Dielectric methods to characterize properties and models to predict structure-property relations at high frequencies above the relaxation frequency (> 10 GHz).	Impedance models and mixing rules to determine dielectric properties at high frequencies for nanoscale single-phase and organic- inorganic hybrid materials. Techniques for assessing nonlinear dielectric and conduction effects at operating voltages. Reliability models and acceleration factors for time-dependent dielectric breakdown.
Enhanced performance – thermal interface materials (IIIh)	ASTM D5470, the guarded hot plate standard test method for measuring thermal resistance of thermal interface materials, does not fully specify the test design, leading to poor correlation of measurements made at various laboratories. A design guide for ASTM D5470 is needed, along with calibration standards for higher resistance materials.	A design guide for ASTM D5470 test method and a set of calibration standards with the same form and approximate thermal performance expected of the thermal interface materials.
Enhanced performance – heat dissipation (IIIi)	Methods to measure thermal conductivity and diffusivity at the nanoscale and correlate the thermal characteristics with defects, interfaces, and anisotropy.	Extended capabilities of current measurement instrumentation, *e.g.,* the widely accepted 3ω technique for direct measurement of thermal conductivity. Appropriate testbeds with controlled defects, interfaces, anisotropy, and 3D geometries to correlate heat dissipation with nanoscale features.
Enhanced performance – structure of multilayers (IIIj)	Precise and repeatable measurements of layer uniformity, thickness, surface roughness, and composition. High-resolution, accurate measurement techniques, fundamental models of the instrumentation and measurement methodology, methods to estimate model and instrument error, and calibration artifacts.	High resolution x-ray reflectometry instrumentation and methodology to measure lattice parameters along with first-principles models to refine the structural parameters. Instrument calibration artifacts.
Enhanced performance – epitaxial layers (IIIk)	Precise and repeatable measurements of layer composition and thickness, crystal defect density, and strain.	High-resolution x-ray diffraction instrumentation and methodology to measure lattice parameters, models to calculate layer composition and thickness, crystal defect density, and strain from lattice parameter measurements, and standards for characterizing the instrumental profile function.
Low *k* Dielectrics (IIIl)	Methods to measure the modulus, hardness, and toughness of 150 nm – 200 nm low-*k* films under various stress states and chemical environments and correlation of these with semiconductor manufacturing yield.	Nanoindentation-based fracture and deformation tests using large acuity probes with supporting analyses and confirmatory MEMS-based tests.

Novel dielectric embedded passive materials with thicknesses less than 500 nm and dielectric constants greater than 2000 at 10 GHz will be needed to increase the operational speed of future devices. However, incorporation of such dielectrics will cause nonlinear electromagnetic effects and time-dependent dielectric breakdown. Identification of candidate materials will require new measurement methods at higher frequencies (> 10 GHz) to evaluate dielectric behavior and its effect on device performance and reliability. (MN IIIg) Finally, the power of integrated circuits increases with decreasing size, so improved thermal interface materials for connecting heat-generating integrated circuits to a heat sink will be needed. Effective dissipation of heat is an increasingly critical issue affecting device reliability and lifetime. Existing techniques for measuring thermal properties are designed for bulk materials or films on substrates and therefore cannot address heat dissipation issues in next-generation devices with complex geometries and nanoscale dimensions. The development of optimized thermal interface materials requires new measurement methods, standards, and models to assess the thermal conductivity, diffusivity, and resistance at the nanoscale. (MN IIIh,i)

Because material properties and, ultimately, device performance, are closely linked to structure, there is a need for high-resolution, accurate measurements to assess uniformity, thickness, surface roughness, and composition of the individual layers in nanoscale, multilayer thin-film structures. (MN IIIj) Additionally, improved measurements are required to evaluate defects and strains in epitaxial, single crystalline layers. (MN IIIk) High-resolution x-ray diffraction and reflectometry methods coupled with instrument calibration artifacts are best-suited to meet these structural measurement needs.

Finally, it is noted that many of the impediments to introduction of new materials into integrated circuits arise in fabrication rather than operation. Semiconductor manufacturing processes impose severe chemical and thermomechanical loadings on materials, introducing defects thereby reducing yield. No-where is this more true than in the introduction of nano-porous low dielectric constant (low-*k*) thin films used in microelectronics interconnection array structures.

Low-*k* materials allow device dimensions to shrink without increasing signal propagation delays and cross-talk between the active semiconducting elements in the device. However, the introduction of porosity to decrease *k*, inevitably leads to decreases in modulus, hardness, and particularly toughness in these materials, which are then unable to withstand the rigors of the manufacturing process. Methods to measure the mechanical properties of low-*k* materials at device dimensions (150 nm – 200 nm film thickness) are required to develop and screen new materials and optimize manufacturing processes, obviating the need for expensive, empirical "build it and see" approaches. (MN IIIl)

<u>Magnetic Storage – Table IV</u>

Future-generation magnetic data-storage devices, tape drives and hard disk drives, with terabits-per-square-inch information densities, require manufacturing and operating tolerances that are beyond the current state of knowledge. Such tolerances reflect the nanoscale feature sizes in the recording head and the nanoscale head-media separations required to achieve such information densities. The ceramic thin-film material sets in the head (amorphous AlO_x, crystalline NiZn ferrites, and $CaTiO_3$) are different from those in microelectronics devices, but forming the nanoscale features in the head has many of the same difficulties as fabrication of advanced microelectronics devices, with the same attendant measurement challenges. However, distinct from their applications in microelectronic devices, the ceramic materials in magnetic storage devices perform crucial mechanical functions during operation to maintain the head-media spacing: In a hard disk drive, the Al_2O_3-TiC composite head body flies over the media on a specially formed air-bearing surface (ABS) that sets the overall head-media separation (~ 10 nm). At the rear of the head, the thin-film stack of the magnetic read-write elements is displaced (~ 1 nm) from the ABS by both irreversible effects from manufacturing and reversible thermomechanical effects during operation. In a tape drive the media moves over magnetic elements centered in the head but the mechanical requirements and effects are the same. Many of the measurement needs for advanced magnetic storage devices are related to the mechanical properties of the ceramic materials used in the head as these set the all-important head-media separation and thus recording density (see Table IV).

In designing recording heads, account must be taken of the deformation of the materials arising from thermal expansion effects on cooling down from thin-film deposition processes during manufacturing and on heating effects during operation. Hence, measurement of the elastic modulus and coefficient of thermal expansion (CTE) of the ceramic materials used in the head is crucial for optimized design and operation. However, as the number of thin-film layers is increased, their structures made more complex, and their thickness reduced to the single nanometer range, the mechanical properties and stability of those film structures are significantly different from their bulk or even microscale counterparts. Current, stylus- or atomic force microscope (AFM)-based approaches are inadequate for measuring mechanical properties at the required length scales as these approaches have reliable response depths of approximately 100 nm. New methods of mechanical characterization are required in order to predict the reversible CTE-mediated head-media separation changes occurring operation. (MN IVa, Table IV)

Superior instrumentation is also required to measure the irreversible separation changes introduced during manufacturing. During formation of the ABS the softer materials of the magnetic thin-film stack that form the actual read-write device recede relative to the ABS. Such pole-tip recession (PTR) also occurs during operation. Accurate measurement of PTR is required to optimize device design, materials selection and manufacturing processes. Measurement of the recess of the poles from the ABS is required, as well as the recess of the structures surrounding the poles. Currently, only the recession of the poles from the ABS is measured but is reported as PTR; the measurements are made using optical interferometry and AFM, without adequate spatial resolution and with inadequate repeatability. Attainable accuracies appear to be the order of three nm. (MN IVb)

Contact between the head and the media causes failure of hard disk drives. Increased head-media friction is an indicator of contact, but there is no known technology for adequately detecting and characterizing head-media interface friction events for next-generation hard disk drives. Current friction-measurement methods lack the required precision, sensitivity, frequency response, and freedom from drift to deal adequately with isolated individual and high-frequency intermittent head-disk contact events. Existing strain gauge technologies are not sufficiently sensitive, are prone to drift and have low bandwidth (1 kHz at best). (MN IVc)

Contact between the head and the media in tape systems also causes failure: Head wear due to abrasion by the tape, which increases the head-tape spacing and signal loss, is a major cause of reduced performance and failure of magnetic tape systems. Crystalline Al_2O_3 particle (~ 200 nm in size) are included in tape media to clear debris from the head, but these, of course, wear the softer materials of the thin-film head stack. Understanding the effects of the media as a basis for controlling head wear and predicting head life is limited by the inability to adequately characterize the abrasivity of the tape. (MN IVd) In addition, the thickness of the protective layer on the tape needs to be measured and controlled beyond current capabilities. This layer influences both head-media spacing and head wear. (MN IVe)

Finally, decreasing the spacing between the magnetic head and the disk medium in a hard disk drive means decreasing the flying height of the head with respect to the medium. However, both the head-medium spacing and the flying height become relatively more uncertain as the spacing decreases. The clearance between the head and the medium depends not just on the physical separation, but on the interactions between the diamond-like coating on the head and the lubricant coating on the medium. As the head-medium spacing decreases from 20 nm in 2003 to

Table IV. Measurement Needs and Solutions for Magnetic Storage

Subtopic (MN #)	Measurement Need	Measurement Solution
Materials selection – mechanical properties (MN IVa)	Techniques to measure mechanical properties such as modulus, hardness, yield strength, and other elastic and plastic properties of thin films.	Indentation and surface probing techniques that measure mechanical properties of the top few nanometers of material with ultra-small force, deformation, and response depth. Calibrated nano-mechanical reference standards for modulus, hardness, yield and shear strength for films thinner than 2 nm.
Materials and process selection – PTR (MN IVb)	Pole-tip recession (PTR) measurement accuracies of less than 1 nm are required now with a measurement target of 100 pm and with associated measurement accuracy of $\pm$ 10 pm. A physical artifact to verify and standardize performance of AFM and optical equipment for measuring sub-nm level PTR.	PTR step standard reference materials. Reference standards (for AFM PTR measurements) that reflect the shape of the anticipated recession and optical properties of the materials. Method for measuring the relative recession of the device with respect to the surrounding poles. Si (111) single atom step height standards (312 pm). Adaptation of such standards to geometries closer to that of a pole tip structure and development of Si (100) standards with heights of about 100 pm
Head-media interactions – friction (MN IVc)	Industry-standard method for the drift-free, high-bandwidth, high-sensitivity measurement of head-disk contact friction compatible with existing test equipment.	Sensor (perhaps MEMS-based) with 1 μN sensitivity, 0.1 μN precision, 1 MHz bandwidth, and less than 0.1% per hour drift that is easy to use, sufficiently robust for daily use by operators and technicians, and self-calibrating.
Head-media interactions – abrasion (MN IVd)	A method that correlates the abrasion property of tapes with the wear of tape heads.	A new method for direct measurement of the abrasivity of tape media.
Head-media interactions – thickness (MN IVe)	An effective and standardized method for measuring the thickness of the protective coating, including the thickness of the lubrication film, on magnetic tape.	Adapt, with compensation for the flexibility of tape, the measurement processes and standards from the hard disk drive industry, in which deposited thin-film, hard-disk media has been used for many years.
Head-media interactions – fly height (MN IVf)	An optical fly height measurement standard that simulates high-density disk fly height conditions and which fits existing fly height testers.	A transparent glass package with a tapered air gap calibrated by phase shifting interferometry and refractometry with a thin end of 2 nm to 5 nm and a thick end of 50 nm to 200 nm.

a projected 2.8 nm in 2013, manufacturers may be able to make relative measurements of the various spacings and do this in a dynamic way, but an absolute calibration and standard is required to determine the function of storage technologies more accurately. (MN IVf)

Lasers and LEDS – Table V

Lasers play a critical role in virtually all technologies, for example, transmission in optical communications systems, data storage and retrieval from compact discs and DVDs, instruments in surgical procedures, and target designators for weapons systems. All solid-state lasers operate by stimulated emission of photons in ceramics, either oxides or compound semiconductors. While there are few measurement needs for mature oxide-based laser systems, measurement developments are critical for semiconducting lasers, particularly short-wavelength blue lasers based on III-nitride materials. Although blue lasers have tremendous potential due to their higher storage density compared to conventional red lasers, widespread application of blue lasers is limited by the ability to accurately and reliably measure laser power and energy. (MN Va, Table V) Measurement tools are also needed to assess the composition, electronic, and optical properties of III-nitride materials at the nanoscale that affect the quantum efficiency, stability, and reliability of the lasers. (MN Vb)

Table V. Measurement Needs and Solutions for Lasers and LEDs

Subtopic (MN #)	Measurement Need	Measurement Solution
Lasers - power and energy (Va)	Accurate, reliable measurements of laser power and energy for low power, continuous-wave blue and UV laser sources and for high-power tunable lasers.	Transfer standards for the wavelength range 260 nm to 400 nm that have linear response up to 20 mW power levels. Primary laser power standards for the laser wavelength range 260 nm to 400 nm. Methods for efficiency, lifetime and reliability testing of sources.
Lasers and LEDs - physical properties (Vb)	Measurement methods to determine (i) carrier transport and recombination mechanisms resolved on the nanometer scale; (ii) alloy composition and phase separation; (iii) quantum efficiency; and (iv) minority carrier diffusion length.	Nanoscale measurement tools for compositional, electronic, and optical property determination in III-nitride materials
LEDs – luminance (Vc)	Methodology to define and rate luminance (light output) of large area LED displays made of discrete devices having non-uniform emission in terms of the emitting area. Measurements must account for the large fluctuations in light intensity when the LED displays are viewed at different angles and distances.	Develop measurement correction factors based on the bright-dark fluctuation.
LEDs - effective intensity (Vd)	Methodology to define effective intensity, a metric that attempts to compare flashing light sources with different flash profiles. Effective intensity data covering the effects of flash profiles, chromaticity, and spectral power distribution.	Measurements of effective intensity for a range of different LED flashing lights. A new effective intensity model based on computational analysis, simulations, and vision experiments.

Light emitting diodes (LEDs), another ubiquitous optical component based on III-V and III-nitride semiconductors, are used in diverse applications such as displays, traffic signals, optical communications, and remote control devices. There are intensive efforts to increase the efficiency of III-nitride LEDs for solid-state lighting, a technology that would result in substantial energy cost savings and reduced environmental impact over current incandescent lighting. Increasing the efficiency requires new measurements to evaluate the relationships between the processing variables, structure of the nitride layers, and optical properties of the LED. (MN Vb) One emerging application for LEDs is large area displays for road signaling, billboard advertising, and stadium displays. Large area LED displays are significantly brighter, more durable, viewable from a larger angular range, and more energy efficient than current self-illuminated display technologies. Development of large area LED displays is hindered by the lack of measurement methods that can account for the large fluctuations in light intensity for different viewing conditions, and a methodology to define and rate the luminance, or light output, of the displays. (MN Vc) Another emerging application for visible light LEDs is flashing light sources to replace rotating beacons or xenon strobes that have limited flash profiles. Methods to assess effective intensity, a metric that attempts to compare flashing light sources with different flash profiles, are needed to commercialize visible LEDs. (MN Vd)

Microelectromechanical Systems (MEMS) – Table VI

The interest in MEMS is rapidly expanding with products such as match-stick size bio-toxin sensors, artificial eyes and the robotic surgical tools to insert them, ubiquitous shock sensors and bright video screens for hand-held devices, or low-cost patches and implants for automated drug delivery on the horizon. Such devices extend MEMS from those with no moving parts (*e.g*., inkjet print heads) and non-contacting moving parts (*e.g*., accelerometers and pressure sensors) to those with contacting moving parts (e.g., digital mirror actuators). This extension drives measurement needs for the small-scale forces associated with contacts and the consequences of such contacts for device performance.

At the system level, the development and commercial introduction of advanced MEMS-based devices is impeded by a lack of measurement techniques to determine, predict, and verify device reliability (device performance over service life). This lack hampers materials and process selection and restrains MEMS devices to the few (rather simple) commercial products for which there are substantial empirical experiences (in restricted environments) of reliability. Without advanced measurement techniques to refine and demonstrate MEMS reliability, customer acceptance of MEMS devices will remain low and MEMS innovators, developers and manufacturers will have little economic incentive to commercialize products. (MN VIa, Table VI)

At the contact level, improved functionality and performance of MEMS devices can only be realized if the imbedded MEMS components move reliably, that is they do not adhere or stick, in a range of operating conditions. Device-scale measurement of adhesion in a range of operating conditions is another critical need. (MN VIb)

At the individual component level, the ability to provide accurate nanoscale force measurements will enable better design of MEMS devices and lead to cost savings from improved tolerances. Atomic Force Microscopy (AFM) is widely used for characterizing surfaces at the nanoscale; however, accurate understanding of the forces being applied to surfaces is limited by the lack of SI-traceable standards at the force scale applicable to AFM. This affects not only specific force measurements (adhesion, deformation, friction) but imaging as well, since excessive forces can lead to structural deformations that alter the image and can lead to erroneous interpretation.

Improved nanoscale measurement accuracy will enhance the ability to measure materials microstructures and properties at the nanoscale and increase our abilities to evaluate and enable MEMS structures. (MN VIc)

Table VI. Measurement Needs and Solutions for MEMS

Subtopic (MN #)	Measurement Need	Measurement Solution
Device reliability (VIa)	Measurement tools for each element of reliability engineering: (i) identification of defects; (ii) measurement of defect kinetics; (iii) development of lifetime models; and (iv) verification of lifetime predictions.	Develop capability to manufacture realistic MEMS test structures (mechanical, thermal, electrical, chemical, fluidic) to enable measurement (in application environments) of the material kinetic properties related to defect appearance. Develop non-empirical lifetime models and the associated analysis software. Develop capability to test many devices rapidly to enable lifetime prediction verification.
Nanoscale adhesion (VIb)	Accurate, device-scale measurement of adhesion with: nanometer positioning accuracy; ability to control contacts at sub-nanometer scales; and applicability to a wide range of materials and temperatures.	Nanoscale adhesion measurement tools and methods that are broadly applicable to MEMS materials and geometries, and which can distinguish the various components of adhesion, including the effects of chemistry and true contact geometry.
Force measurement - standards (VIc)	Accurate nanoscale force measurements on elements of MEMS devices.	Reliable standards for the calibration of AFM force measurements: Uniform reference cantilevers with stiffness values relevant to AFM cantilever spring constants. The cantilevers must be calibrated, with measurements traceable to the SI, and have a standardized measurement procedure.
Force measurement - standards (VId)	Easily transported small force standards with which to calibrate instruments such as AFMs and nanoindenters.	Development of transportable small force standards based on accurate characterization of atomic and molecular forces.

Energy Generation – Table VII

Energy conservation and reduction of fossil fuel emissions are top priorities for the Nation, so intensive efforts are underway to develop alternative energy sources and systems. Three classes of ceramic materials are strong candidates for energy applications in the 21st century—thermoelectrics, high temperature superconductors, and solid oxide fuel cell materials. Measurement needs and solutions for each of these materials classes are summarized in Table VII.

Thermoelectric materials, *i.e.,* materials that can directly convert thermal energy into electrical energy, and vice-versa, show great promise for vehicular waste heat-recovery and solid-state refrigeration applications. The ability to recycle automotive waste heat (~75% of the energy content of each gallon of gasoline) into electricity to run a hybrid motor on the vehicle would greatly increase efficiency. Solid-state refrigeration applications offer the possibility of “green” refrigeration, and also greater reliability due to the absence of moving parts. Existing thermoelectric materials suffer from low energy conversion efficiencies, so new materials with greater figures of merit $ZT = \alpha^2\sigma/\lambda$ are needed (where α is the Seebeck coefficient, σ is electrical conductivity, and λ is thermal conductivity). Bi_2Ti_3 and other tellurides are the most well-known thermoelectric materials, but there is an intensive effort to discover higher ZT materials, such as skutterudite (*e.g.,* $CeFe_3CoSb_{12}$) and Chevrel (*e.g.,* $(Cu,Fe,Ti)Mo_6Se_8$) phases. Identification of promising materials will require high-throughput methods to measure ZT, particularly for thin films, which currently have the highest reported ZT values. (MN VIIa, Table VII)

Hydrogen or natural gas-powered fuel cells convert chemical energy to electrical energy directly and are a promising means of providing clean power to small, distributed sites such as vehicles or residences. Solid oxide fuel cells employ multiple ceramic materials as the working components (anode, cathode and electrolyte) of the cell and operate at temperatures in excess of 1000 ºC. The high temperatures and chemical complexity of the reactions at the interfaces of the

Table VII. Measurement Needs and Solutions for Energy Generation Systems

Subtopic (MN #)	Measurement Need	Measurement Solution
Thermoelectrics – properties (VIIa)	Measurement methods for reliably determining ZT, particularly for thin-film materials, and standards to calibrate measurements. A high-throughput approach for screening of thermoelectric thin films is also needed to accelerate materials optimization.	A scanning thermoelectric microscopy technique, based on scanning tunneling microscopy, would allow relatively fast property screening of combinatorial film libraries. Standardized measurement procedures and related reference materials for ZT measurements.
Fuel cells – properties (VIIb)	In-line, real-time measurement methods to rapidly assess the mechanical, electrical, and chemical properties of the fuel cell and its components.	Optical and x-ray based methods to measure properties of fuel cells during production to provide fundamental correlations between component fabrication tolerances, assembly tolerances, and overall fuel cell performance.
Fuel cells – performance (VIIc)	Test methods to measure the performance of residential fuel cell systems in a variety of sizes, types, and operational strategies. Predictive models to enable rapid rating of fuel cell performance.	A standard test method to determine residential fuel cell performance at various environmental, electrical, and thermal loads. A model to assess fuel cell performance from a limited number of laboratory experiments.
Fuel cells – hydrogen probes (VIId)	*In situ* hydrogen-sensitive probes for (i) real-time imaging of fuel cell devices during operation; and (ii) characterization of the behavior of novel fuel cell materials.	Powerful, non-destructive neutron-based probes with micrometer to nanometer spatial resolution and customized sample geometries to (i) obtain real-time tomographic images of fuel cell devices; and (ii) monitor hydrogen in highly neutron-absorbing and/or increasingly complex fuel cell materials.
High temperature superconductors – properties (VIIe)	Methods to measure the properties of long lengths of wires at high currents, low temperatures, and high electro-mechanical forces.	Variable-temperature critical-current measurements methods, bi-axial tape bending instrumentation, low temperature delamination test techniques, and variable-angle, high-magnetic-field strain testing methods.

different materials makes it challenging to characterize the chemical, electrical and mechanical properties. Widespread commercialization of fuel cells is hindered by a lack of understanding of how these properties affect fuel cell performance under different operating conditions. In particular, in-line real-time measurements are needed to assess the properties of fuel cells during manufacture. (MN VIIb) Test methods are also needed to rate the performance of fuel cells under various environmental conditions and thermal and electrical loads. (MN VIIc) Tools to visualize hydrogen transport in candidate fuel cell materials and operating fuel cells would provide important information for the selection of optimal fuel cell materials, designs, and operating conditions. (MN VIId)

Twenty years after their discovery, ceramic high temperature superconducting materials in the form of wire for cabling and tape for motors are poised to revolutionize the electric power industry. Existing superconducting power grid prototypes are expensive, have limited current density capability, and are not mechanically robust. In contrast, yttrium barium copper oxide superconductors have good mechanical integrity and offer 5 to 10 times as much performance and potential cost reduction. However, manufacturing these second-generation wires in sufficiently long lengths for commercial use hinges on the ability to measure properties along the full length. (MN VIIe)

Cutting Tools

By increasing machining speeds to the highest practical level, time is saved and costs are reduced. In machining, the maximum cutting speed is limited by the temperature generated at the tool tip, so measuring the cutting temperature is a vital step in obtaining the optimum speed for any particular machining process. Temperature measurements also are essential for verifying or validating modeling and simulation methods employed in the quest for improved efficiency. Measurement of temperature in the cutting zone of a machining process is challenging because the temperature gradients are high, and therefore, extremely high spatial resolution (on the order of one micrometer) is needed to map the temperature in the tool and work piece. Further, the machining process is highly dynamic (strain rates of 10^3 s^{-1} or higher) requiring high temporal resolution. A practical barrier in measuring temperatures in machining experiments is the difficulty in obtaining a clear view of the very narrow shear zone (less than one mm). The challenge is thus to measure the temperature fields in a small, mm-scale, zone with gradients of 1000 °C through the zone. The most promising methods for such measurements are those employing state-of-the-art infrared or thermal cameras.

Civil Infrastructure – Table VIII

Concrete is the primary material for the Nation's civil infrastructure (bridges, pipes, pavements, foundations, office buildings, residential houses) and must have adequate long-term (*e.g.,* decades) performance. Increasing the durability of concrete will significantly extend the lifetime of concrete structures, resulting in significant cost savings. Innovation in concrete and cement products is limited by the lack of suitable test methods and methodologies to measure long-term performance. Methods are needed to identify the specific physical, chemical, and mechanical mechanisms that contribute to long-term durability and predict the lifetime of ceramic products. (MN VIIIa, Table VIII)

Ceramics are widely used as fire-resistive materials to protect steel structures, particularly tall buildings. Currently, mineral fiber and gypsum-based materials are spray-coated onto steel structures for fire protection. Next-generation materials such as foamed concrete and intumescent coatings can greatly increase passive fire protection, allowing more time for evacuation and enhanced protection of the structure. A robust materials-science-based measurement infrastructure is needed to evaluate the durability and fire resistance of these advanced materials. This infrastructure should include reliable, standardized test methods for assessing performance, and structural and fire dynamic models to predict performance. (MN VIIIb)

Advanced insulation materials including ceramic-based powders, foams and glass fibers are being developed for refrigeration applications. The thermal resistance of these materials is an order of magnitude greater than that of conventional insulation materials, so use of the advanced materials in refrigeration products will result in greater flexibility in product design and significant cost reductions due to lower energy consumption. The ASTM C518 Standard Test Method for evaluating the thermal performance of most insulation products uses a calibration standard; however, the upper limit of thermal resistance value for this standard is lower than the resistance values of the advanced materials. The thermal resistance of the advanced materials must be accurately defined to assess the overall performance of products incorporating the materials. Thus, new certified calibration standard materials with higher thermal resistance values are needed to bring the advanced materials into products. (MN VIIIc)

Table VIII. Measurement Needs and Solutions for Civil Infrastructure Systems

Subtopic (MN #)	**Measurement Need**	**Measurement Solution**
Concrete – durability (VIIIa)	Methods to identify the degradation mechanisms limiting durability. Improved accelerated test methods to predict durability.	Test methods and long-term performance rating methodologies developed by consensus standards organizations. Predictive models of durability for relevant environmental conditions.
Fire-resistant materials (VIIIb)	Measurement techniques to quantitatively characterize the three-dimensional microstructure so that links between microstructure and performance can be identified and exploited. Measurement methods to quantify the thermal conductivity and adhesion properties, specifically at high temperatures.	X-ray microtomography methods to quantify three-dimensional microstructures. New, standardized measurement techniques for determining the necessary thermophysical and adhesion/mechanical properties.
Thermal insulation – test methods (VIIIc)	Thermal performance test methods with increased accuracy for insulation products.	Development and production of calibration standards with thermal resistance values comparable to those associated with advanced insulation products.

Pervasive Measurement Needs – Table IX

Measurement needs concerning the handling of information related to materials and product development, whether that information is measured, simulated, or perceived, extend across all industrial sectors. At the largest level, and the one that exerts the greatest influence on materials development, handling of materials data itself is a measurement need: Competitiveness and innovation are strongly dependent on timely access to reliable data and computational models. Assembling, refining, and accessing the extensive amounts of materials data so as to create, or simply find, materials information is the subject of the emerging field of materials informatics. Materials informatics is an enabling technology that can control the rate of development and implementation in any materials sector. The impact, therefore, is not strictly a matter of the productivity that materials informatics enhances, but rather the control or dominance of whole market sectors. Traditionally, distinct aspects of materials informatics have been addressed separately, resulting in many severe issues and barriers relating to the interoperability of diverse data resources, the compatibility of data and computational resources, the ability to interrogate databases via automated remote access, and the suitability of linked resources for discovery of trends and relationships. The ultimate goal may be considered the dynamic integration of materials selection, property data access (with specified temperature and stress conditions and level of reliability), and computational models (with real time, adaptable boundary conditions) into an iterative, product-model optimization cycle. (MN IXa, Table IX)

An example of a measurement need associated with the materials informatics of experimental data is that of phase diagrams. The technical feasibility of next-generation applications frequently depends on the availability of ceramics with enhanced or entirely new properties, while commercial feasibility depends on cost reduction. This combination creates pressure (in industrial, academic, and government materials research labs) to find ceramic materials with distinct properties and reduced production costs. Every deliberate effort to discover new materials or to improve material processing begins with the knowledge of what works now, or almost works, particularly regarding chemical composition and the conditions under which the materials are stable. For many ceramics, including some already in commercial use, the paucity or complete lack of relevant, reliable data is an impediment to research and development, cost reduction, and successful implementation of innovative materials. Phase equilibrium data provide the essential physicochemical information pertaining to chemical composition and the conditions of temperature and pressure under which pure compounds (and mixtures thereof) are thermodynamically stable. However, once such data are generated, materials informatics approaches are required to merge the new data with the existing wealth of phase diagram data and then access the new extended data set to obtain information regarding development directions. (MN IXb)

Materials informatics approaches are also required to accurately model and simulate the behavior of new products, processes, or materials across all industrial sectors. This is particularly so in nanomaterials development, for which there is a lack of linked computational models of objects at the nano-, micro-, and larger scales and a lack of reconciliation of the new nanoscale measurement technologies and computational models. A major contributor to the poor performance of models simulating behavior of innovative products, processes, or materials is the lack of a unified informatics scheme to provide and assure the quality of the data required to support decisions that affect the deployment of new materials and processes in manufacturing. Preliminary information about the risks of new technologies, for example nanotechnology, has been highly sensationalized, yet no effort to systematically collect, assess, and measure the data needed for risk analysis has been made.

Finally, there are measurement needs associated with perception. An example is that color and appearance guides nearly all consumer product purchases, often being the first attribute noticed in a store display or advertisement. Appearance matching is critical for successful manufacturing, particularly in a global economy where components of a product are manufactured at different sites for later assembly at a single site. Using the current methods of visual inspection, rejection rates of the final products are on the order of 50%. Better color measurement and detection can significantly reduce rejection rates. The development of instrumentation to absolutely quantify surface appearance or to quantify the degree to which materials or objects match based on surface appearance is challenged by the interplay between visual perception, color, lighting, material optical properties, and surface texture. Cosmetics, coatings, clothing, food, automobiles, and appliances are some of the products for which proper measurement of color and appearance by manufacturers and suppliers is critical for commercial success. (MN IXc)

Table IX. Pervasive Measurement Needs and Solutions Across all Technology Sectors

Subtopic (MN #)	Measurement Need	Measurement Solution
Materials informatics (MN IXa)	A materials informatics scheme that is objective, robust, and scientifically-sound that is based on consensus of the methods and standards for measuring materials data and assuring their quality.	Provision of an effective IT infrastructure for the collection and dissemination of all relevant materials data. Expert system software realizing conformance with physical and chemical laws and utilizing robust statistical evaluation algorithms.
Phase equilibrium data (MN IXb)	Phase diagram data that are available in a readily accessible and usable database.	Measure, gather, evaluate, and disseminate phase equilibrium data as part of an integrated materials informatics effort.
Visual perception (MN IXc)	Measurement techniques to correlate appearance and visual perception.	High-resolution instruments for complete characterization of surface scattering and the determination of an optimal set of measurements (spatial, spectral, and angular) necessary to capture, specify, and render surface color and appearance, including texture.

IV. CONCLUSIONS

In examining the survey here on the measurement science and technology required for ceramics innovations, conclusions may be drawn on examining the measurement needs as a whole, on comparing groups of selected measurement needs, and in considering the specifics of individual measurement needs. We address each level of aggregation in turn.

A view of the survey as a whole shows that there is no single concept defining a measurement need. Measurement needs exist in the intuitive areas of tangible physical characterization and measurement (*e.g.*, toughness measurements for low-*k* dielectric films), the related advanced instrumentation (*e.g.*, high-precision x-ray diffractometers for strain measurement in epitaxial layers), and extend to the development of completely new science and instrumentation (tip-enhanced scanning Raman spectroscopy for strain measurement). Measurement needs also exist in the intangible areas of information handling for both high-throughput testing (*e.g.*, as in combinatorial methods for assessing barrier-layer effectiveness in integrated circuits) and in the materials informatics field, in which large amounts of data from a large number of sources are assembled, validated, and sorted (*e.g.*, as in exploring the phase diagrams of new materials). Finally, measurement needs may also exist in the form of tangible standards (*e.g.*, an air gap wedge for assessing the fly height of a magnetic storage head) or as intangible standards (*e.g*, the physico-chemical assessment cascade protocol for biomedical nanoparticles). Diverse as the concept of a measurement need is, however, all the measurement needs here either are directed towards assessing the performance of a device or product (*i.e.*, how well it performs an intended function, say the strength/weight ratio of a nanotube-based composite) or measuring a material property used in defining a metric of performance (say the thermal conductivity included in the *ZT* metric of a thermoelectric heat exchanger).

Many measurement needs extend across industrial sectors. This commonality is perhaps best examined via the familiar materials science and engineering sequence of processing-structure-properties-performance. For example, in the area of processing, high-throughput testing methodologies, and the combinatorial analyses to handle the consequent large amounts of test data, are required in generating phase diagrams, optimizing the composition of thermoelectric cooling and heat-recovery materials, assessing the effectiveness of diffusion barriers, and selecting workable combinations of high-*k* gates and contact electrodes for integrated circuits. In the area of structure, atomic-scale, three-dimensional maps of material composition and structure are required to identify limiting defects in blue lasers and to assess the path of hydrogen through fuel cells. At larger scales, rapid measurements of small-scale device dimensions are required for self-assembled structures, integrated circuit particle contamination, and magnetic storage recording heads. In the area of properties, adhesion and mechanical properties measurements at small scales are required for MEMS and magnetic storage devices, nanoparticles, and nanotubes-containing composites. Measurement methods of thermal properties of composite materials or structures are required for integrated circuits, thermal insulation for advanced refrigeration or building fire protection, and cutting tools. Finally, in the area of components performance, measurement methods for assessing and predicting environmental effects on reliability and lifetime are required for concrete infrastructural elements, MEMS devices, *in vivo* behavior of nanoparticles, and magnetic tape head wear. Many other examples can be found in the small sample of measurement needs discussed here. This suggests that perhaps a better way to deploy resources to address measurement needs overall within the USMS is by the property to be measured or the performance metric to be optimized, rather than by the perceived needs of a particular industrial sector. For example, the microelectronics industry has significant mechanical and thermal properties measurement needs that other industrial sectors, perhaps apparently needing more directly, also share. Another example might be that the MEMS and concrete industries both have performance measurement needs in reliability and lifetime testing and modeling.

Finally, we note that much of the measurement science and technology, and the related measurement needs articulated here as examples, extends across all material classes—metals, ceramics, polymers, and their composites—and is certainly not restricted to ceramics. In fact, this survey has taken a deliberately broad view of what constitutes a ceramic in identifying measurement needs required for ceramics innovations. The term ceramic has been interpreted as applying to materials and structures in which the "ceramic" is just one of many components, *e.g.*, activated nanoparticles, nanotube composites, polymer-matrix composites, or data-storage tape; or is not traditionally regarded as a ceramic, *e.g.*, high-*k* and low-*k* dielectric thin films, thermoelectric and photonic compound semiconductors, or silicon-based MEMS materials. This broad interpretation is suggested by, and suggests, two ideas that are crucially important for the advancement of ceramics innovations: The first is that the intellectual skill set developed over the last 100 years of ceramic science and engineering is directly applicable to many of the materials under development today for advanced products and devices. The skill set was based on the characteristics of ceramic materials—predominantly ionic-covalent bonding, usually a compound, limited reactivity, small conductivities, and tendency to brittleness. Many "modern" materials also have these characteristics. Hence, the ceramics community—through application of its skill set—is extremely well-positioned to contribute to the measurement science and technology required for modern materials innovations. Adjustments are required to recognize the applicability of ceramics concepts to materials that are not formed by high-temperature processes, that are nanoscale, and which are used in combination with many other "non-ceramics," but the underlying science remains the same.

The second idea deriving from our consideration of measurement needs for ceramic innovations is that it is extremely difficult to separate out a ceramic measurement need from that of other materials, or of a material or component that contains many materials in combination. Modern materials science and engineering has developed over the last 100 years from optimizing the properties of monolithic materials (largely through microstructural manipulation) to optimizing the performance of components and devices by fabricating structures using materials in combination. Hence, development of measurement science and technology, which enables a ceramic material to be incorporated into a device and thus enables new functionality, cannot solely focus on the ceramic material, may not even involve the ceramic material, and may be broadly applicable to many other material types as well. Many of the measurement needs identified here fall into this last category: Although discussed in the context of an enabling ceramic technology, the required measurement techniques, instrumentation, standards, and informatics extend across all material classes. Many of these materials-independent needs are driven by innovation at the nanoscale, for which distinctions between the traditional, macroscopic, materials classes are not very meaningful.

Hence, in identifying the underlying measurement science and technology that enables ceramics innovations it is useful to recognize that measurement needs extend beyond science and instrumentation to standards and informatics, that individual measurement needs extend across technology sectors, and that the great wealth of ceramic science and technology is applicable to many materials classes.

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