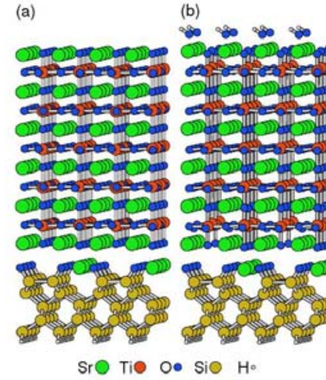
National Institute of Standards and Technology
William A. Jeffrey
Director

Technology Administration
Michelle O'Neill
Acting Under Secretary of Commerce and Technology

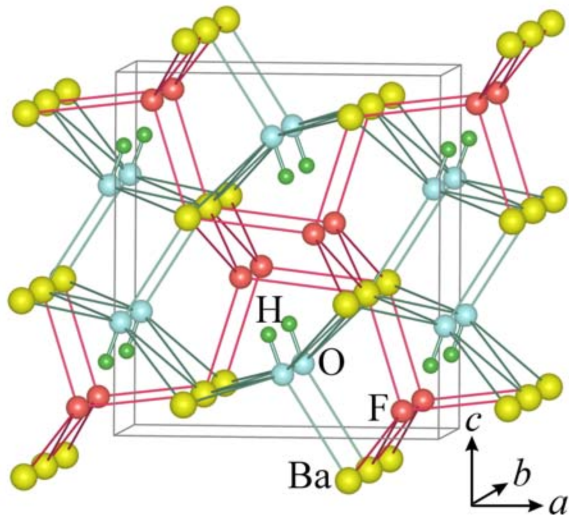
U.S. Department of Commerce
Carlos M. Gutierrez
Secretary



MATERIALS SCIENCE AND ENGINEERING LABORATORY

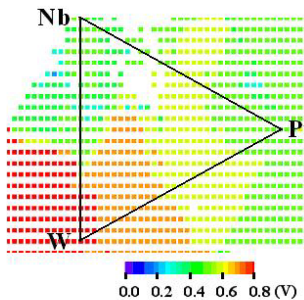
FY 2005 PROGRAMS AND ACCOMPLISHMENTS

Ceramics Division



Debra L. Kaiser, Chief

Ronald G. Munro, Deputy



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Ceramics Division

It has been an exciting year in the Ceramics Division for forging new partnerships with industry and other national laboratories, initiating research efforts relevant to emerging nanomaterial systems, and strengthening our ongoing core metrology, data, and standards activities closely aligned with the mission of NIST. Our accomplishments predominately relate to Nanometrology and Materials for Electronics, two of the five program areas in the Materials Science and Engineering Laboratory (MSEL). To crown these achievements, our excellent technical staff members have been honored with a host of prestigious NIST and external awards.

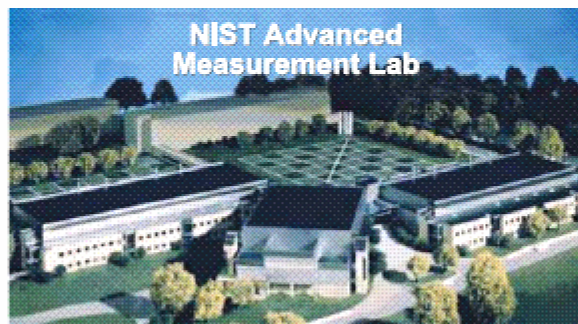
A concerted effort to partner directly with Sematech has led to research activities in advanced metrology to address pressing needs in next-generation semiconductor products. One project is aimed at developing metrology for accurate thin film characterization using x-ray reflectometry, while the other is focused on applying novel combinatorial and synchrotron methods to optimize the interfaces in advanced high-k dielectric CMOS gate stacks critical to the semiconductor industry's 21st century technology roadmap.

A partnership with the National Cancer Institute (NCI) was initiated this year. NCI has awarded NIST a three-year research grant to collaborate with NCI's new Nanotechnology Characterization Laboratory in the development and application of nanoparticle-based systems for cancer prevention, detection, and therapeutics. The Ceramics Division is contributing its long-standing expertise in nanoparticle metrology to this effort by developing measurement methods and protocols for characterizing the size, size distribution, and dispersion of inorganic and organic nanoparticles in aqueous solutions compatible with body fluids.

The Ceramics Division has maintained its strong commitment to two outstanding long-term partnerships aimed at providing the reliable, high-quality data that is fundamentally essential for advanced technology research and development: the celebrated NIST–American Ceramic Society collaboration on phase equilibria diagrams and the collaboration with FIZ Karlsruhe (Germany) on the renowned FIZ–NIST Inorganic Crystal Structure Database.

Ongoing partnerships at DOE synchrotron user facilities have continued to provide high-quality, unique capabilities for structural and chemical characterization of advanced materials. In a joint effort with Sandia National Laboratory, considerable progress has been made to establish a synchrotron-based variable kinetic energy XPS facility at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory (BNL), the first of its kind in the U.S. For the fifth consecutive year, the soft x-ray beamline jointly developed by NIST and Dow Chemical Co. was the most productive soft x-ray facility at NSLS, yielding over 25 publications. Our long-term partnership in UNICAT, a collaborative access team at the Advanced Photon Source (APS), Argonne National Laboratory, has continued to support and improve numerous scattering and diffraction techniques. This partnership will undergo a transition next year as operation and management of the beamline will be assumed by the APS.

The Ceramics Division has continued to support the upgrade and expansion of its unique measurement capabilities. Instruments in the high-resolution x-ray metrology and nanotribology facilities in the NIST Advanced Measurements Laboratory (AML) became fully operational this year and have already yielded results with unprecedented resolution. With the recent modernization of two beamlines at NSLS dedicated to extended x-ray absorption fine structure (EXAFS) and x-ray photoelectron spectroscopy (XPS), the Ceramics Division and its partners have established the capability to perform x-ray absorption spectroscopy spanning all elements in the periodic table. A three-year SBIR project has led to the development of a state-of-the-art multi-element detector at the NSLS soft x-ray beamline, providing an order of magnitude increase in data collection rates.



We represent MSEL in the AML, the world's premiere metrology laboratory.

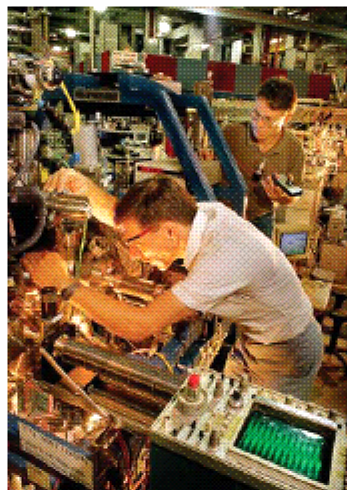
There have been numerous notable scientific achievements across the Division this year; following are several representative research highlights. Ten international leaders worked together to create the IUPAC–NIST Crystal Phase Identifier standard to uniquely identify any chemical compound appearing in an electronic database. This landmark standard is a major step forward towards the worldwide interoperability of crystal structure databases. In collaboration with BNL scientists, our unique near-edge x-ray absorption fine structure metrology facility at the NSLS has been employed to characterize the surface order and structure of carbon and boron nitride nanotubes,

resulting in four refereed journal publications. A feature article reviewing the state-of-the-art in characterizing ceramic materials by x-ray and neutron small-angle scattering was published in the *Journal of the American Ceramic Society* (A. Allen, *J. Am. Ceram. Soc.*, 88, 1367, 2005). The first computational model to correlate nanoscale chemical ordering, defects and properties in relaxor ferroelectrics, materials of choice for sonar and medical imaging transducers, was constructed, coupling first principles calculations and molecular dynamics simulations. Devices for calibrating force in commercial nanoindentors, instruments for measuring mechanical properties at the nanoscale used by thousands of researchers worldwide, have been developed jointly with scientists in the NIST Manufacturing Engineering Laboratory. These force calibration cells will be produced in collaboration with the major nanoindentor instrument manufacturers.

In a far-reaching effort to respond to the anticipated metrology and standards needs for next-generation advanced materials, particularly for nanotechnology applications, several new research efforts were initiated this year. In the area of CMOS technology, measurement methods are being developed and applied to evaluate thermal and electrical stability at interfaces in high-k dielectric gate stack structures and to characterize the electronic structure and chemical bonding in these structures at nanometer depth sensitivity. Metrology based on Raman spectroscopy and x-ray topography is under development to evaluate the stress state and defect structures in strained silicon layers for high-performance MOSFET devices.

Multifunctional oxide materials, wherein the functional response of one constituent phase/subsystem is generated by the response of another phase/subsystem to an external field, offer the potential for integrating electronic, magnetic and optical devices on a single chip. A combined experimental/theoretical modeling effort is underway to analyze the formation of self-assembled epitaxial nanostructures of multifunctional oxides, measure the functional properties at the nanoscale, and ultimately correlate the responses to the nanostructural architectures. Foundational pre-standards research on theoretical structural models for extracting film properties from x-ray reflectometry measurements and on reference cantilevers for calibrating AFM force measurements has begun. A multi-year project was initiated to develop an *in situ* nanocalorimetry technique with adequate sensitivity to detect hydrogen desorption in hydrogen storage materials and interfacial reactions in multilayer structures in collaboration with world leaders in nanocalorimetry from the University of Illinois.

It is a pleasure to acknowledge the numerous prestigious honors bestowed upon the staff this year. Dr. Daniel Fischer was one of only twelve individuals to receive the coveted Arthur S. Flemming Award honoring outstanding Federal Government employees. Dr. Fischer was cited for his pioneering work in developing and utilizing a first-in-the-world facility for soft x-ray absorption spectroscopy that has enabled key scientific and technological advances in cutting-edge and emerging technologies of paramount importance to the Nation. For these exemplary achievements, Dr. Fischer was also awarded the Department of Commerce Gold Medal, the highest honorary award granted by the Secretary of Commerce.



Dr. Daniel Fischer received the Arthur S. Flemming Award and the Department of Commerce Gold Medal award.

The Silver Medal, the second highest Department of Commerce honorary award, was given to a NIST team, including Dr. Douglas Smith, for their technical innovation that revolutionized the realization of the unit of force at the micro- and nanoscales. Dr. Vicky Karen and Dr. Alec Belsky (Technology Services) received the Bronze Award, the highest honorary recognition presented by the NIST Director, for their development and application of scientific algorithms and functional software embodied in the Inorganic Crystal Structure Database used for phase identification in commercial SEMs.

The esteemed NIST Edward Uhler Condon Award recognizing distinguished achievement in scientific and technical writing was awarded to Dr. Ronald Munro for his eloquent and systematic exposition of data evaluation as a scientific discipline. Nearly 1500 printed and 40,000 electronic copies of NIST Recommended Practice Guide "Data Evaluation Theory and Practice for Materials Properties" have been requested across the world, attesting to the broad appeal and applicability of the work.

We remain committed to providing high-quality metrology tools, standards, and data to support the development and implementation of advanced ceramic materials, components, and devices in the electronics, photonics, energy, and healthcare technology sectors.

Debra L. Kaiser
Chief, Ceramics Division

Advanced Measurement Capabilities

The special role that NIST plays in measurement metrology and standards often results in the development of instruments and facilities, as well as measurement methods, capable of probing material characteristics in ways unavailable elsewhere. These capabilities often provide the context and observations necessary to the development of a fundamental understanding of the mechanisms of material behavior. The Ceramics Division is privileged to have an excellent staff and world leaders who are responsible for the design and development of many of these unique instruments and facilities.

Debra L. Kaiser

Nanometrology

Working with instrument makers, the Nanotribology Group, directed by Dr. Stephen Hsu, has designed and built a remarkable facility featuring a cluster of equipment and instrumentation that establishes a new state of the art for conducting measurements and observations of nanoscale events. Included in this facility are a nanoadhesion apparatus, a dual white-light interferometer microscope, a multiscale friction tester, and a multifunction apparatus combining an ultrahigh vacuum scanning tunneling microscope with an atomic force microscope (UHV STM/AFM). Together, these instruments allow us to image, manipulate, and measure adhesion, friction, and other nanomechanical properties of materials from microscale to nanoscale dimensions.

Nanoadhesion Apparatus

Adhesion is an important issue in nanotechnology as well as in device manufacturing industries. Conventional adhesion measurement relies on large interfacial areas and the accurate positioning of two surfaces. Force measurements typically are performed by strain gauges and transducers with force sensitivities in the newton and millinewton ranges. These conventional instruments are unsuitable for nanoscale contacts. To resolve this issue, the newly designed NIST apparatus, Figure 1, allows adhesion force measurements between two surfaces at the nanoscale level using the AFM force measurement principle. In this device, capable of nanonewton force resolution, a laser light is piped through a fiber-optic cable directed at a quadrant photodetector. Because the length and stiffness of the fiber can be modified easily, a wide range of spring constants can be obtained, and the measurement of adhesion forces can be achieved at multiple force levels. Thus, the NIST apparatus has established an exciting new opportunity to measure the influence of surface forces on adhesion, molecular interactions, and the compliance of surfaces, all critical data to device manufacturing industries.

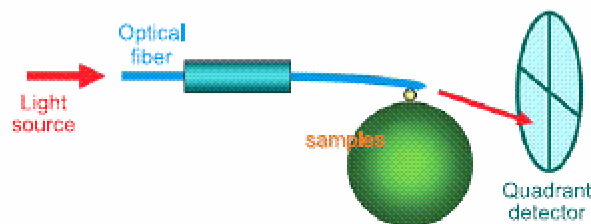


Figure 1: Schematic operation of the NIST nanoadhesion apparatus.

UHV STM/AFM

Enhanced by the superior vibration isolation and clean room environment in NIST's new Advanced Measurement Laboratory (AML), our UHV STM/AFM system, Figure 2, provides atomic imaging and force measurement with an unprecedented resolution and accuracy.

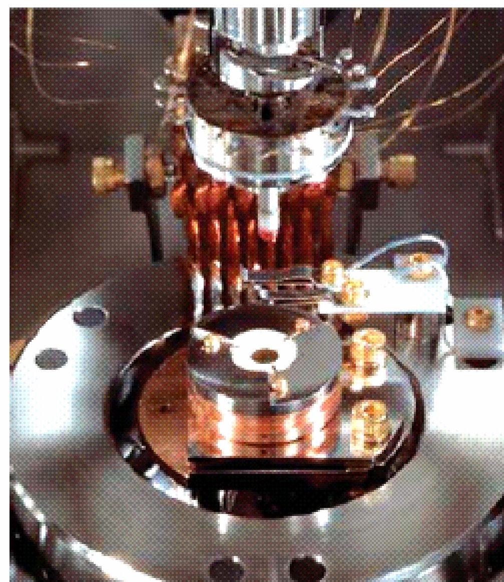


Figure 2: Ultrahigh vacuum STM/AFM apparatus.

Combined with our conventional AFM, equipped with a "triboscope" attachment, we are able to image and manipulate surface features and measure a wide range of material characteristics and properties important in nanodevice operation. As a result, we now are leading fundamental metrological efforts, working with device

and magnetic hard disk industries, as well as academic institutions, to establish reliable measurement methods, calibration artifacts, and infrastructural support for current and future industries.

Multiscale Friction Tester

At the nanoscale level, friction between movable components is a significant concern. To address this issue, NIST and Hysitron, Inc. have collaborated to develop a three-dimensional, capacitance based, force sensor. The stiffness and position of the probe tips can be tightly controlled to yield accurate information of the contact area. Force measurements can be made in the range from 100 nN to 1 N in the z-direction, and 500 nN to 0.5 N in the x,y-directions. Displacements can be measured from 0.2 nm to 5 µm in the z-direction, and 10 nm to 15 µm in the x,y-directions. Consequently, accurate friction measurements can be conducted at length scales ranging from the nanometer scale to the micrometer scale. Already, this instrument has enabled us to resolve apparent disparities in nanofriction measurements and to understand the nature of what had been thought to be anomalous friction behavior. (See Friction Scaling in the Technical Highlights section of this report.)

High Resolution X-ray Metrology

Powder and single crystal x-ray diffraction are widely used in industry, research facilities, and academia as one of the principal means of characterizing materials. Both techniques yield a wealth of information on the crystallographic and microstructural character of the specimen. The powder diffraction method has the virtue that it can probe a continuous sequence of crystallographic reflections with a single scan in angular space, while single crystal methods can be adapted to high resolution analysis of materials such as thin films.

However, results from both powder and high-resolution techniques are affected by a complex optical aberration function that is specific to the diffraction optics and goniometer assembly used in the experiment. NIST Standard Reference Materials (SRMs) are the recognized means by which these aberrations may be characterized to achieve improved measurement accuracy. To address these issues, Dr. James Cline of the Data and Standards Technology Group conceived and designed the Ceramics Division Parallel Beam Diffractometer (CDPBD).

The CDPBD, Figure 3, was designed and built specifically to perform traceable measurements on powder and thin film specimens. Installed in the new NIST Advanced Measurement Laboratory, this facility provides the environmental and temperature controls requisite for a new generation of NIST SRMs that will enable unprecedented measurement accuracy in a highly competitive, data conscious, materials research community.

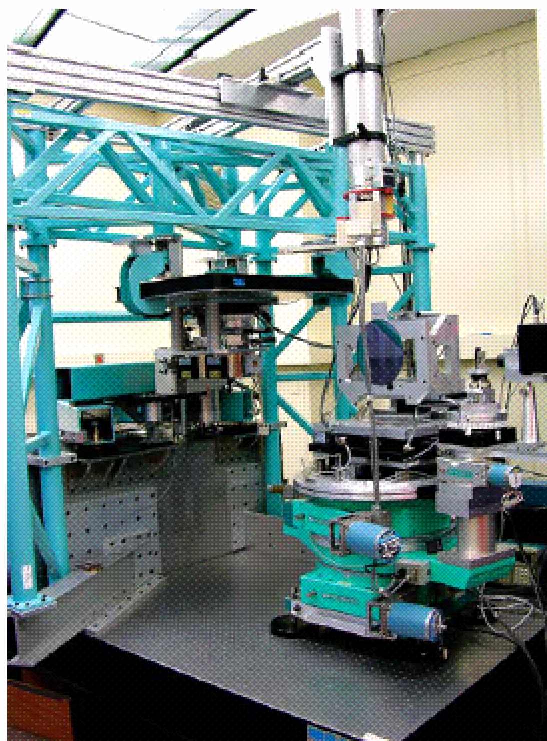


Figure 3: The Ceramics Division Parallel Beam Diffractometer in the AML.

X-ray Absorption Spectroscopy

Advances in our x-ray absorption spectroscopy facilities have achieved an unrivaled capability enabling our Characterization Methods Group (CMG) to address a remarkably broad range of challenging structure and chemistry issues at the forefront of materials science research today. Through the application of a truly unique combination of beamline facilities, we are able to examine characteristics of surfaces, interfaces, and bulk materials in a manner heretofore inaccessible.

To achieve this capability, CMG, led by Dr. Daniel Fischer, brought together a suite of three unique high-throughput x-ray spectroscopy beamlines (designated U7A, X24A, and X23A2). Housed in the National Synchrotron Light Source located at Brookhaven National Laboratory in New York, these beamlines, taken together, can easily examine nearly all of the naturally occurring elements in the entire periodic table. This year, the capabilities of beamline U7A, used for soft x-ray materials science applications, were significantly enhanced by the addition of a 14 element, state-of-the-art, Si (Li) fluorescence yield detector, providing best in the world resolution. Further enhancement of U7A, Figure 4, was achieved through the installation of a 6-axis manipulator that enables the exciting prospect of molecular alignment studies.

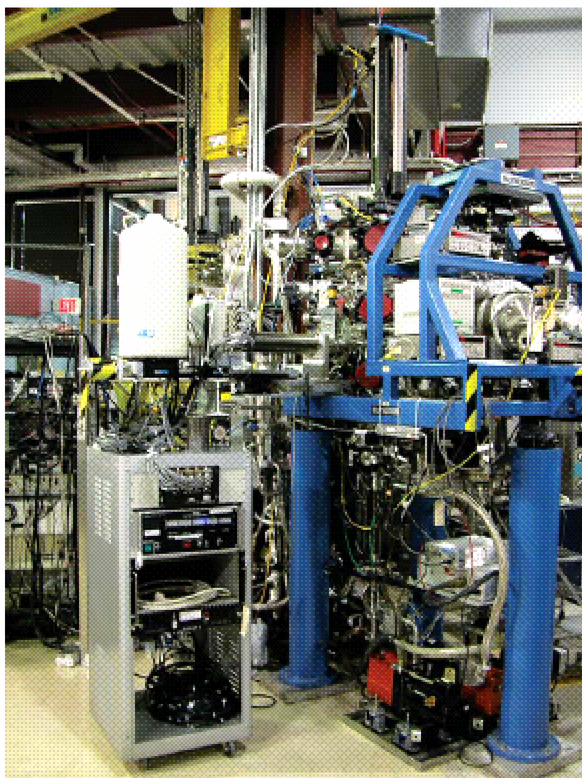


Figure 4: The soft x-ray spectroscopy station on beamline U7A.

Building on these capabilities, CMG has initiated a long-term plan, co-funded with Sandia National Laboratory, for establishing a variable energy XPS (x-ray photoelectron spectroscopy) and NEXAFS/EXAFS (near-edge/extended x-ray absorption fine structure) scientific program utilizing beamline X24A. A new, fully automated materials science end-station is planned, modeled after the very successful high throughput attained on U7A. The emphasis for this work will be on the use of variable energy XPS for chemical depth profiling, sub-surface chemistry, and interface chemistry.

X-ray Topography

The premier, dedicated, monochromatic topography facility in the U.S. has been developed and implemented at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL), Argonne, Illinois. X-ray topography is used to study the defect microstructure of single-crystal materials by imaging the diffracted intensity from selected lattice planes of the sample. The NIST effort, led by Dr. David Black, takes advantage of the third-generation synchrotron x-ray source to provide a spatially enlarged beam, up to 140 mm x 50 mm, with large-area real-time imaging detectors to perform static and dynamic experiments with large samples. In addition to topography, radiographic and phase contrast imaging are available. This instrument has recently been used to study subsurface damage in 300 mm production silicon wafers, defects in thin strained-silicon films, strain around nanoindentations and dendrite growth in Sn/Bi alloys. In the case of the strained-silicon films, by using grazing incidence geometry we have been able to directly image defects in a film ≈ 165 Å thick. These defects have been shown to mimic the misfit dislocation structure in the underlying relaxed Si/Ge virtual substrate. Understanding how the substrate defects propagate into the strained-silicon film is a critical issue in the development of the next generation of strained silicon electronic devices.

Ultrasmall-Angle X-ray Scattering

Our ultrasmall-angle x-ray scattering (USAXS) instrument, constructed as part of the UNICAT facility, is housed at the APS at ANL. The NIST effort is led by Dr. Andrew Allen. The facility provides the most versatile SAXS facility for materials science and engineering research in the world. Using a single instrument configuration, the representative microstructures within a wide range of heterogeneous materials of technological importance can be quantified over a scale range extending from nanometers to micrometers. The continuously tunable x-ray energy enables anomalous SAXS studies to be made, allowing different microstructural components within a composite system to be distinguished. Microstructures also can be measured when they possess an arbitrarily high anisotropy, such as those encountered in some coating materials and membranes. Recently, the high APS brilliance has been combined with the instrument's high-precision x-ray crystal optics to provide a spatial resolution on the order of 10 µm for resolving the microstructural gradients within solid oxide fuel cell (SOFC) components.

The USAXS instrument serves microstructure metrology needs in a wide class of technological problems encountered in metals and alloys, structural ceramics, thermal barrier coatings, fuel cells, biological scaffolds, flowing nanoparticle suspensions, soot in flames, and polymer gels. Furthermore, the intrinsic absolute intensity calibration provided by this instrument underpins our partnerships with others to develop specialized SAXS instrumentation for interrogating surface structures in electronic gate materials, or to approach sub-micrometer spatial resolution in SOFC research.

For More Information on this Topic

S. Hsu, J. Cline, D. Fischer, J. Woicik, D. Black, A. Allen (Ceramics Division, NIST)

Nanometrology

Nanotechnology will revolutionize and possibly revitalize many industries, leading to new and improved products based on materials having at least one dimension less than 100 nm. The federal government's role in realizing the full potential of nanotechnology is coordinated through the National Nanotechnology Initiative (NNI), a multi-agency, multi-disciplinary program that supports research and development, invests in a balanced infrastructure, and promotes education, knowledge diffusion, and commercialization in all aspects of nanoscale science, engineering, and technology. NIST's unique and critical contribution to the NNI is *nanometrology*, defined as the science of measurement and/or a system of measures for nanoscale structures and systems. NIST nanometrology efforts focus on developing the *measurement infrastructure* — measurements, data, and standards — essential to advancing nanotechnology commercialization. This work provides the requisite metrology tools and techniques and transfers enabling measurement capabilities to the appropriate communities.

MSEL plays a vital role in nanometrology work at NIST with efforts in four of the seven NNI Program Component Areas — *Instrumentation Research, Metrology and Standards for Nanotechnology; Nanomaterials; Nanomanufacturing;* and *Fundamental Nanoscale Phenomena and Processes.* Innovative projects across MSEL are defining and addressing the forefront research issues in these areas.

Instrumentation Research, Metrology and Standards for Nanotechnology

R&D pertaining to the tools needed to advance nanotechnology research and commercialization. The design, development, and fabrication of nanodevices will require nanomechanical measurements that are rapid, accurate, predictive, well-understood and representative of a device or system's environment in real time. MSEL is addressing this need by developing instrumentation, methodology, reference specimens and multi-scale modeling approaches to quantitatively measure mechanical properties such as modulus, strength, adhesion, and friction at nanometer-length scales. This year, novel instruments for measuring adhesion and friction forces between surfaces and nanoparticles were developed jointly with industrial partners. Quantitative maps of elastic modulus were obtained by innovative methodologies based on atomic force microscopy and strain-induced elastic buckling instability. To address the need for quantifying measurements made with widely-used commercial nanoindentors and scanned probe microscopy instruments, MSEL is developing reference specimens and SI-traceable force calibration methodology.

Nanomaterials

Research aimed at discovery of novel nanoscale and nanostructured materials and at a comprehensive understanding of the properties of nanomaterials. Among the many classes of nanomaterials, nanotubes have received great attention due to their remarkable physical properties relevant to many applications. In response to needs expressed by industry and other federal agencies, MSEL has embarked on a new effort to develop a suite of metrologies and standards aimed at characterizing key structural features and processing variables of carbon nanotubes. These include dispersion, fractionation, orientation, alignment, and manipulation of individual single-walled nanotubes, all critical to establishing efficient bulk processing schemes to meet the imminent high demand for carbon nanotubes.

Nanomanufacturing

R&D aimed at enabling scaled-up, reliable, cost-effective manufacture of nanoscale materials, structures, devices, and systems. Nanoimprint lithography (NIL) is rapidly emerging as a viable high-throughput technique for producing robust structures with a patterning resolution better than 10 nm. MSEL is developing metrologies that are crucial to advancing NIL as an industrial patterning technology for the electronics, optics, and biotechnology industries. The current focus is on characterizing shape and the fidelity of pattern transfer, two key factors in achieving widespread commercial application of NIL.

Fundamental Nanoscale Phenomena and Processes

Discovery and development of fundamental knowledge pertaining to new phenomena in the physical, biological, and engineering sciences that occur at the nanoscale. The magnetic data storage industry needs the ability to measure and control magnetization on nanometer length scales and nanosecond time scales to meet increasing demands for reduced size and increased speed of devices. MSEL is developing measurement techniques to elucidate the fundamental mechanisms of spin dynamics and damping in magnetic thin films. Work this year has focused on measurements of the effects of interfaces and interface roughness on magnetization dynamics and magnetic characterization of edges in magnetic devices.

Through these and other research activities, MSEL is maintaining its committed leadership in developing the measurement infrastructure for current and future nanotechnology-based applications.

Contact: Debra L. Kaiser (Ceramics Division)

Mechanical Metrology for Small-Scale Structures

Industrial trends toward miniaturization require quantitative mechanical property data for design, development, and fabrication of modern small-scale devices. Developments in disruptive technologies require engineering materials data for structures and architectures at multiple (nano to macro) length scales. Accordingly, we are designing and developing mechanical testing configurations from small-scale in-situ *structures for localized measurements of fracture and deformation behavior of materials and interfaces.*

Edwin R. Fuller, Jr. and Douglas T. Smith

This project aims to: (i) measure mechanical properties of microstructures for myriad industrial and biological systems that cannot be fabricated in bulk samples; (ii) study small-scale phenomena, which may be controlled by surface effects (*e.g.*, the influence of surface stresses on crack nucleation and extension); and (iii) obtain quantitative mechanical property data of materials and interfaces for designing small-scale structures and components and for assessing their mechanical reliability. Well-characterized testing configurations are being designed and developed for measurements of strength and crack extension of small-scale structures and interfaces. We are pursuing four tasks: (i) configuration design, optimization, and characterization via finite-element analyses; (ii) specimen fabrication; (iii) mechanical testing and fracture analysis (fractography); and (iv) length and force metrology. In addition to work in the Ceramics Division (tasks i, iii, and iv), two collaborations were established in the fabrication task (ii): one with James A. Beall of the Quantum Electrical Metrology Division in NIST Boulder, and one with Northwestern University.

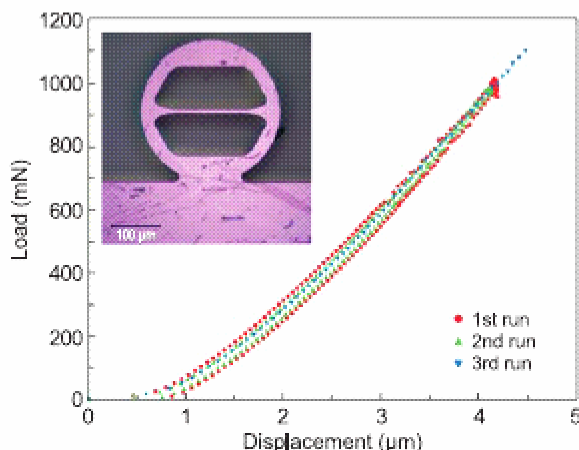


Figure 1: Repeatability for multiple loading of a single specimen.

Significant progress has been made in the design of a compressively loaded test configuration with a well-defined, tensile gage section. The inset of Figure 1 shows one such specimen fabricated by James Beall using deep reactive ion etching (DRIE) of a silicon wafer. When a load is applied to the top of this theta-like geometry, a uniform uniaxial tensile stress develops in the middle gauge section. Finite element analysis gives (horizontal) gauge section stress and strain as functions of the applied load and load-point displacement, respectively. Dimensionless calibration factors have been obtained for several specimen and fillet geometries, and as a function of the gauge width.

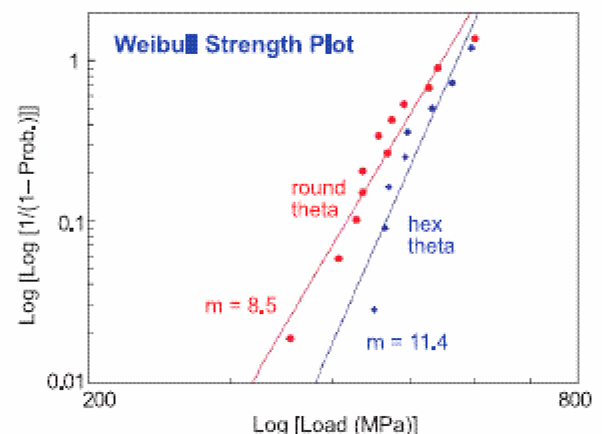


Figure 2: Weibull plots for round and hexagonal configurations.

Specimens are tested using a depth-sensing nanoindenter as a universal testing machine, thereby giving a continuous record of applied load and load-point displacement. Repeatability is illustrated in Figure 1. Strength data for DRIE silicon, Figure 2, suggest that differences for round and hexagonal specimen configurations are not significant.

To extend this technique to a wider variety of material systems, focused-ion-beam (FIB) milling is being explored in collaboration with Northwestern University. Hexagonal theta specimens fabricated from a lamellar directionally solidified eutectic of $Ni_{0.5}Co_{0.5}O$ and ZrO_2 are approximately 1/15 the size of the silicon specimens.

Contributors and Collaborators

D. Xiang, G.D. Quinn, D.L. Henann (Ceramics Division, NIST); J.A. Beall (Quantum Electrical Metrology Division, NIST); N. Alem, V.P. Dravid (Northwestern University)

Nanotribology and Surface Properties

Accurate determination of adhesive and frictional forces between surfaces and particles is critical for efficient and effective design and development of nanoscale devices and manufacturing processes. Working with diverse industrial partners (instrument, device, and magnetic storage industries), we are addressing this critical need by developing metrology tools and methods for nanomechanical property measurements.

Stephen M. Hsu and Richard S. Gates

One of the major conclusions from the National Nanotechnology Initiative workshop on instrumentation and metrology (held at NIST in January 2004) was need for improved tools, methods, and calibration procedures for nanoscale measurements. Several advances in measuring friction and adhesion and controlling surface lubrication and texturing were achieved this year.

Advances in Instrumentation

Existing atomic force microscopy (AFM) and multiscale friction testing instruments were upgraded to improve measurement accuracy and extend applicability of the methods. The AFM was extensively modified to increase the signal-to-noise ratio. A joint effort with Hysitron resulted in a new 3-D force sensor to conduct friction and scratch tests with much higher accuracy. Sample stage modulation is being implemented across several platforms to increase sensitivity and expand measurement capability. In-house cantilever and tip fabrication capability and collaborations with numerous specialty tip fabricators were established.

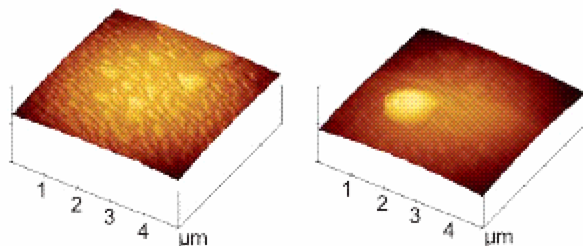


Figure 1: Surface features on colloidal probes.

In adhesion and friction measurements, surface forces are critical parameters that depend upon the real area of contact. Figure 1 illustrates typical colloidal probes showing random surface features. A computational procedure was developed to estimate the bearing area for this type of probe enabling better determination of contact areas.

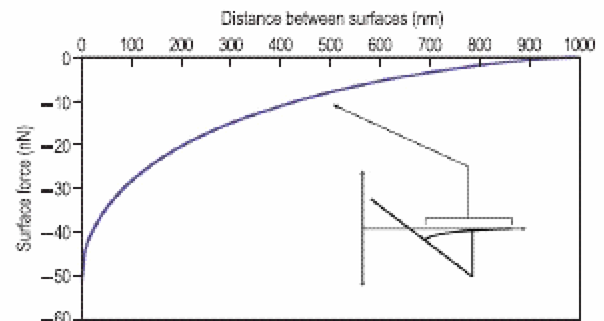


Figure 2: Surface force measurement down to 1 nm. The inset shows a schematic of a typical force curve from the AFM.

The first nanoscale probe using an ultra-thin sheet of mica glued on a colloidal probe was successfully developed to measure surface forces on extremely small areas (Figure 2). To avoid snap-on of the probe tip during approach, the cantilever stiffness and signal-to-noise ratio were increased.

Surface Control

Advances were made in the organization of mixed molecules on surfaces for hydrophobicity, anti-adhesion, and friction control properties. Ultra-durable hydrophobic films and friction control films were demonstrated last year. Collaboration with Dan Fischer at the NIST beam line at the National Synchrotron Light Source continues to be vital in characterizing these complex molecular mixtures.

Surface textures are increasingly being used to control surface energy, polarity, adhesion, and friction. In work supported by other agencies and industries, the surface properties of materials were controlled by use of specific surface features such as dimples, triangles, and ellipses at micro- and nanoscale dimensions.

Interactions

Ongoing interactions with domestic and international partners included a cantilever force calibration study with other National Measurement Institutes and Jon Pratt (NIST, MEL), and surface texture research with seven other countries.

Contributors and Collaborators

C. Ying, S. Yang, M. Reitsma, D.-I. Kim, Y. Liang, X. Wang, J. Grobelny, D. Fischer, Y.T. Hsia (Seagate); W. Gerberich (University of Minnesota); O. Warren (Hysitron); C. Su (Veeco); D. Mendel (NPL); E. Santner (BAM)

Chemistry and Structure of Nanomaterials

Successful nanoscale materials fabrication is empowered by a detailed knowledge of the chemistry and structure of surface bound molecules; e.g., the optimization of SAMs, molecular templates, MEMs lubricants, and functionallized nanotubes. We develop, demonstrate, and advance cutting-edge synchrotron metrologies to bring nanoscale materials phenomena to practical applications.

Daniel A. Fischer, Vincent A. Hackley, and Andrew J. Allen

In potential MEMs lubricants, we have found that the degree of surface ordering in self-assembled monolayers (SAMs) governs the friction properties of the film. n-Alkyltricholorosilanes films with different chain lengths (Cn films where n=5−30) were characterized by near-edge x-ray absorption fine structure (NEXAFS), Fourier transform infrared spectroscopy (FTIR), and atomic force microscopy (AFM). The chain lengths having 12, 16 and 18 carbon atoms were found to be highly oriented with a preferential molecular orientation of the polymeric C-C chains perpendicular to the surface. C5 and C30 SAMs did not exhibit preferential orientation of the alkyl chain and C10 showed partial ordering. Complementary FTIR tudies were done to estimate order qualitatively by peak positions of asymmetric CH2 and the symmetric CH2 stretches. The molecular order information from FTIR followed similar trends as determined by NEXAFS. The frictional properties of the organic monolayers were determined through the simultaneous measurement of normal (load) and lateral (friction) interfacial forces with AFM. Friction measurements on different chain lengths follow inverse trends with surface order from NEXAFS as shown in Figure 1.

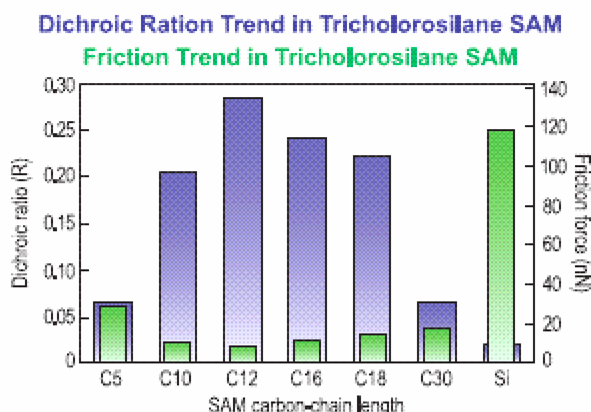


Figure 1: SAM order and friction versus chain length.

The flow-cell developed by NIST for *in situ* ultra-small-angle x-ray scattering (USAXS) studies of solution-mediated nanoscale materials has been applied to the technologically important case of a homogeneously precipitating solution of nanosize ceria (n-CeO_2). n-CeO_2 has multiple applications in catalysis, as a solid oxide fuel cell electrolyte material, and in a number of other areas.

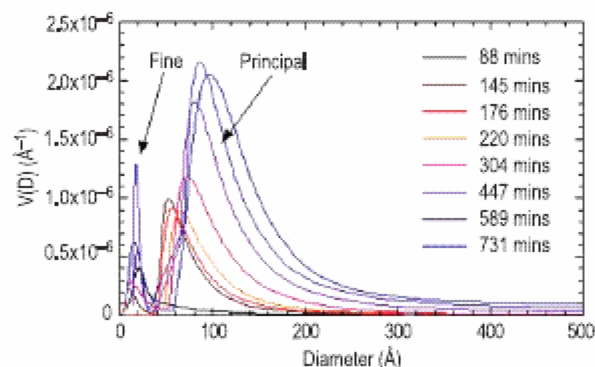


Figure 2: Homogeneous precipitation of n-CeO_2.

Modeling of *in situ* USAXS data taken during reaction at 25 °C indicates a co-precipitation of solid principal particles (see Figure 2) and a population of fine particles with a core-shell morphology. The principal population grows in size and volume fraction, V, but the fine secondary features grow only in volume fraction. It has been postulated that the fine features constitute a step in the formation of the principal particle population, a theory currently being tested with additional experiments in conjunction with Columbia University collaborators.

A strong temperature affect is seen: at 35 °C, the volume fraction of principal particles increases with a growth rate roughly twice that at 25 °C. The activation energy for the n-CeO_2 precipitation was estimated from USAXS data to be about 46 kJ mol^{-1}. Additional studies with the flow-cell focus on dispersion and flow-induced alignment of carbon nanotubes in various solvent/dispersant systems.

Contributors and Collaborators

S. Sambasivan, S. Hsieh, S. Hsu (Ceramics Division, NIST); P.R. Jemian (University of Illinois); J. Ilavsky (APS XOR); S.-W. Chan (University of Columbia); J. Randall, M. Banesh (Zyvex Inc.); H. Wang (Michigan Tech.)

Materials for Electronics

The U.S. electronics industry faces strong international competition in the manufacture of smaller, faster, more functional, and more reliable products. Many critical challenges facing the industry require the continual development of advanced materials and processes. The NIST Materials Science and Engineering Laboratory (MSEL) works closely with U.S. industry, covering a broad spectrum of sectors including semiconductor manufacturing, device components, packaging, data storage, and assembly, as well as complementary and emerging areas such as optoelectronics and organic electronics. MSEL has a multidivisional approach, committed to addressing the most critical materials measurement and standards issues for electronic materials. Our vision is to be the key resource within the Federal Government for materials metrology development and will be realized through the following objectives:

- Develop and deliver standard measurements and data for thin film and nanoscale structures;
- Develop advanced measurement methods needed by industry to address new problems that arise with the development of new materials;
- Develop and apply *in situ* as well as real-time, factory floor measurements for materials and devices having micrometer to nanometer scale dimensions;
- Develop combinatorial material methodologies for the rapid optimization of industrially important electronic and photonic materials;
- Provide fundamental understanding of the divergence of thin film and nanoscale material properties from their bulk values;
- Provide fundamental understanding, including first principles modeling, of materials needed for future nanoelectronic devices.

The NIST/MSEL program consists of projects led by the Metallurgy, Polymers, Materials Reliability, and Ceramics Divisions. These projects are conducted in collaboration with partners from industrial consortia (*e.g.*, SEMATECH), individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs within the government such as the National Semiconductor Metrology Program (NSMP). Materials metrology needs are also identified through the International Technology Roadmap for Semiconductors (ITRS), the International Packaging Consortium (IPC) Roadmap, the IPC Lead-free Solder Roadmap, the National Electronics Manufacturing Initiative (NEMI) Roadmap, the Optoelectronics Industry Development Association (OIDA) Roadmap, and the National Magnetic Data Storage Industry Consortium (NSIC) Roadmap.

MSEL researchers from each division have made substantial contributions to the most pressing technical challenges facing industry, from new fabrication methods and advanced materials in the semiconductor industry, to low-cost organic electronics, and to novel classes of electronic ceramics. Below are just a few examples of MSEL contributions over the past year.

Advanced Gate Dielectrics

To enable further device scaling, the capacitive equivalent thickness (CET) of the gate stack thickness must be 0.5 nm to 1.0 nm. This is not achievable with existing SiO_2/polcrystalline Si gate stacks. High dielectric constant gate insulators are needed to replace SiO_2, and metal gate electrodes are needed to replace polycrystalline Si. Given the large number of possible materials choices for the gate dielectric/substrate and gate dielectric/metal gate electrode interfaces, the MSEL Ceramics Division is establishing a dedicated combinatorial film deposition facility to study the complex interfacial interactions. This same methodology is applicable to a wide variety of problems in the electronic materials field.

Advanced Lithography

Lithography is the key enabling technology for the fabrication of advanced integrated circuits. As feature sizes decrease to sub-65 nm length scales, challenges arise because the image resolution and the thickness of the imaging layer approach the dimensions of the polymers used in the photoresist film. Unique high-spatial resolution measurements are developed to identify the limits of materials and processes for the development of photoresists for next-generation lithography.

Advanced Metallization

As the dimensions of copper metallization interconnects on microelectronic chips decrease below 100 nm, control of electrical resistivity becomes critical. The MSEL Metallurgy Division is developing seedless deposition methods that will simplify thin-film processing and result in film growth modes that increase trench filling, thus lowering interconnect resistivity.

Mechanical Reliability of Microchips

One of the important ITRS challenges is to achieve effective control of the failure mechanisms affecting chip reliability. Detection and characterization methods for dimensionally constrained materials will be critical to the attainment of this objective. Scientists in the MSEL Materials Reliability Division are addressing this issue by focusing on electrical methods capable of determining the thermal fatigue lifetime and mechanical strength of patterned metal film interconnects essential to microchips.

Contact: Martin L. Green (Ceramics Division),
Eric K. Lin (Polymers Division)

Multifunctional Electronic Ceramics

Materials which exhibit exploitable and coupled responses to multiple external fields (electric, magnetic, etc.) present entirely new design opportunities for multifunctional, miniaturized devices such as sensors or actuators. The goal of this project is to establish transferable engineering principles for multi- and single-phase electronic ceramics which exhibit a functional response by one constituent phase/subsystem that is generated by the response of another phase/subsystem to an external field.

Igor Levin, Julia Slutsker, and Terrell A. Vanderah

The development of next-generation multifunctional devices now includes a search for materials exhibiting simultaneous magnetic, electronic, and/or photonic responses (*e.g.*, magnetic semiconductors, magnetic superconductors, multiferroics, etc.). Multiferroic materials display a coexistence of ferroelectric and ferromagnetic responses and have attracted particular interest for several novel device applications including memories, sensors, and actuators. Self-assembled, epitaxial heterophase nanostructures consisting of both ferromagnetic and ferroelectric phases represent one promising class of multiferroics. The strong magnetoelectric coupling obtained in these materials is attributed to the nanoscale distribution of the phases, which facilitates highly efficient elastic interactions and a strong magnetic response to an electric field (or vice versa) via magnetostriction and the piezoelectric effect. The same elastic interactions also control the self-assembly of the component phases, so that the architecture and the scale of the nanostructures can be predicted and controlled by manipulating the stress state of the film.

We analyzed the affect of stress conditions on the morphologies of epitaxial, self-assembled nanostructures using $PbTiO_3$-$CoFe_2O_4$ thin films. The two-phase nanostructures were grown on single-crystal $SrTiO_3$; the strain conditions were varied by deposition on differently oriented substrates. Regardless of orientation, the nanostructures consisted of vertical columns of ferromagnetic $CoFe_2O_4$ dispersed in a ferroelectric $PbTiO_3$ matrix, or vice versa (Figure 1). However, the morphologies of these columns and their spatial arrangements exhibited a marked dependence on substrate orientation. Phase field modeling of these nanostructures, which assumed an equilibrium corresponding to the minimum of elastic and interfacial energies at a given phase fraction, succeeded in reproducing the morphological differences (Figure 2). The modeling confirmed that these differences are related to elastic anisotropy in the film. Our results, which demonstrate that the architecture of self-assembled multiferroic nanostructures can indeed be controlled by a careful choice of the stress conditions, open a tantalizing opportunity for the rational design of self-assembled, multifunctional nanostructures.

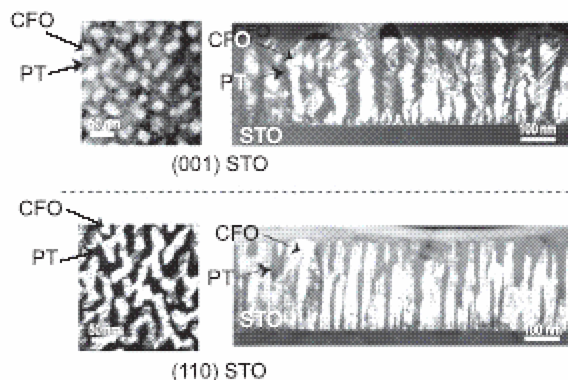


Figure 1: Plane view and cross-sectional images of the $0.33CoFe_2O_4$-$0.67PbTiO_3$ nanostructures grown epitaxially on (001) and (110) $SrTiO_3$.

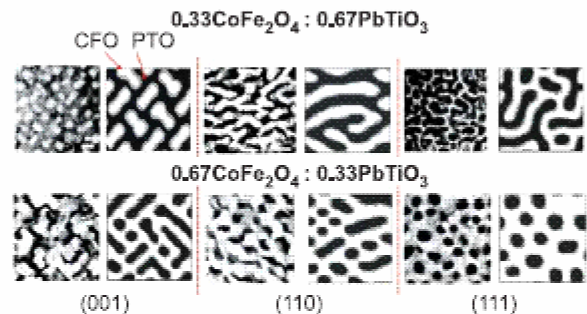


Figure 2: Plane view of $CoFe_2O_4$-$PbTiO_3$ nanostructures grown epitaxially on (001), (110), and (111) $SrTiO_3$. For each composition and orientation, the experimentally observed structure is shown on the left and the simulation is shown on the right.

Research on bulk single-phase materials included phase equilibria studies of the Bi_2O_3-Fe_2O_3-Nb_2O_5 and Bi_2O_3-Mn_2O_3-Nb_2O_5 systems. Surprisingly, both systems feature extensive pyrochlore-type phase fields at compositions requiring mixing of the magnetic ions with far larger Bi^{3+} ions on the A-sites, in apparent violation of traditional substitutional rules. The pyrochlore phases exhibited relative permittivities ~150, and were readily deposited on Si as crystalline thin films using pulsed laser deposition. Although the multiferroic phase $BiFeO_3$ was found to participate in ambient-pressure phase assemblages, $BiMnO_3$ did not, and was not stabilized by the presence of Nb^{5+}.

Contributors and Collaborators

P.K. Schenck (Ceramics Division, NIST); V. Provenzano (Metallurgy Division, NIST); J. Li, A.L. Roytburd (University of Maryland)

Spectroscopy, Diffraction, and Imaging of Electronic Materials

We are working to develop, establish, and provide synchrotron-based metrology, including instrumentation and technical expertise, for spectroscopy, diffraction, and imaging techniques applicable to the study of nanoscale and other phenomena that are important in the design, application, and performance of electronic materials.

Joseph C. Woicik

The epitaxial growth of oxides on silicon opened the possibility of incorporating many of their unique electronic properties into silicon device technology. We have studied the epitaxy and lattice expansion of $SrTiO_3$ thin films grown coherently on Si(001) by kinetically controlled sequential deposition. Coherent growth is achieved by repetition of the deposition sequence that includes a low-temperature and high-oxygen partial-pressure step followed by a high-temperature and low-oxygen partial-pressure step, thereby suppressing the detrimental oxidation of the silicon substrate.

Unlike films grown by more traditional molecular-beam-epitaxy (MBE) methods, these films are found to have an in-plane lattice constant that is indistinguishable from the silicon substrate, an out-of-plane lattice constant that is expanded by an amount twice that predicted by the bulk elastic constants of $SrTiO_3$, and a critical-thickness behavior beyond ~ 2 nm (5 unit cells or 5 monolayers (ML's)).

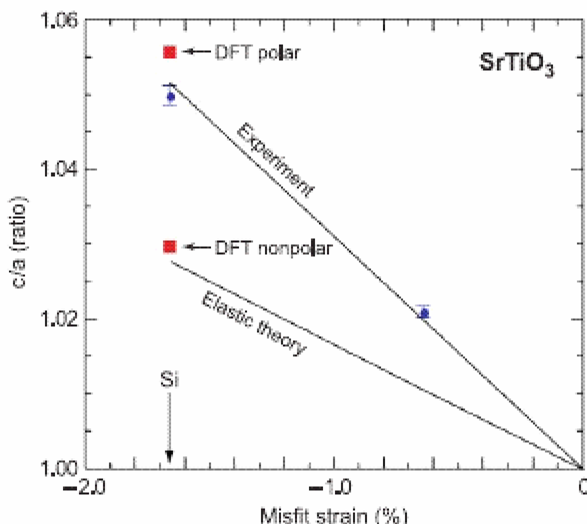


Figure 1: Measured c/a ratio for 5 ML and 10 ML films as a function of in-plane lattice mismatch. Also shown are the predictions of elastic theory and density functional theory (DFT).

The experimentally determined *c/a*-ratio as a function of in-plane misfit strain is shown in Figure 1, for both 5 ML and 10 ML films. The experimental results are compared to the results of density functional theory for the *c/a* ratio of a 5 ML film in coherent registry with the silicon substrate as shown in Figure 2. The difference between the two structures in Figure 2 is the presence of OH adsorbates on the surface and oxygen vacancies at the interface, as revealed by high-resolution x-ray photoelectron spectroscopy for films that have been exposed to air.

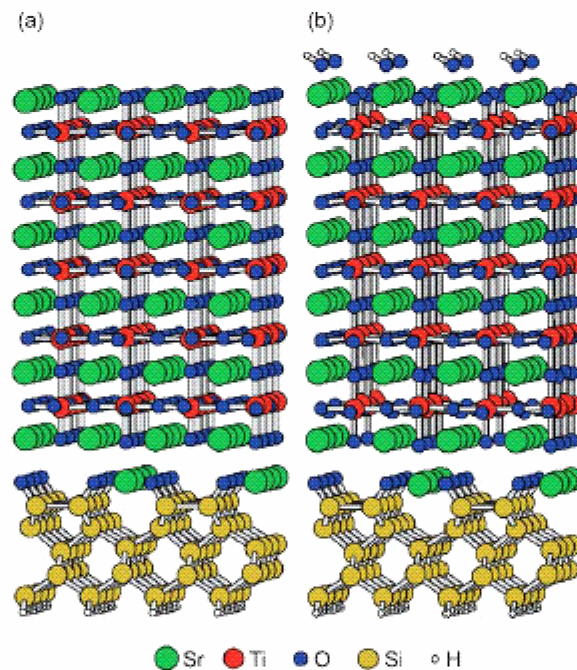


Figure 2: Structure of the ideal 5 ML $SrTiO_3$/Si(001) system (a) and the system with O vacancies and OH adsorbates (b). Note the ferroelectric polarization in (b) but not in (a).

This energetically favorable interfacial-defect/surface-charge structure compensates the ferroelectric depolarization field and allows the ferroelectric polarization in these ultra-thin films that is confirmed by Ti *K*-edge x-ray absorption fine-structure measurements.

Contributors and Collaborators

H. Li (Motorola Labs); P. Zschack, E. Karapetrova (UNICAT); P. Ryan (Ames Lab); C.R. Ashman, C.S. Hellberg (NRL); A. Allen, D. Black, M. Green, I. Levin (Ceramics Division, NIST)

Theory and Modeling of Electronic Materials

Optimizing the properties of electronic materials requires fundamental understanding of the origin of their useful properties and computational tools that connect atomic-scale knowledge obtained from first-principles calculations to properties that emerge at larger length scales. We are developing first-principles based modeling tools for calculating the physical properties of materials as a function of chemical ordering on different length scales and how these properties respond to changes in the environment (temperature, pressure, electric field, etc.). Such techniques have applications in a variety of areas, including dielectrics, dilute spin glass magnets, light-emitting diodes, multiferroics, phase diagrams, piezoelectrics, semiconductors, and spectroscopy.

Eric J. Cockayne, Richard J. Wagner, and Benjamin P. Burton

Understanding and predicting the physical properties of solid solutions is a difficult problem in general. Even in the simplest case of a harmonic crystal, the vibrational energy as a function of atomic arrangement can have a strong affect on the phase diagram. The calculated maximum temperature for the miscibility gap in NaCl-KCl is reduced by about 50 % when vibrational entropy is included, and the agreement with the experimental phase diagram is much improved. Many systems with useful electromechanical properties, such as $PbMg_{1/3}Nb_{2/3}O_3$ (PMN), the relaxor ferroelectric that is a constituent of the ultrahigh piezoelectric compound PMN-$PbTiO_3$, are highly anharmonic. Effective Hamiltonian (H_{eff}) techniques have been developed to simplify the modeling of these systems, but the effect of chemical disorder on the lattice dynamics of solid solutions is not well understood. We published a methodology, based on maximum localization, for automatically determining the appropriate H_{eff} basis for solid solutions and showed that it correctly reproduces the phonon density of states for low-frequency phonons. In PMN, these phonons were determined to be of two types: those involving displacements of Pb, and those involving rotations of oxygen octahedra.

Experiments suggest that the relaxor behavior in $PbSc_{1/2}Nb_{1/2}O_3$ (PSN) is associated with nanoscale chemically ordered regions in a disordered matrix. We performed a molecular dynamics simulation of a PSN H_{eff} on a 320000 atom cell. Polarization fluctuations are much larger in the chemically ordered regions,

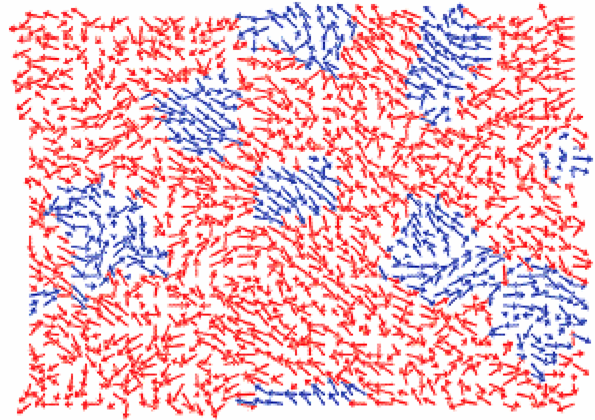


Figure 1: Snapshot of local polarization in PSN model showing larger polarization and polarization correlations in the chemically ordered regions (blue) than in the disordered matrix (red).

which thus dominate the dielectric behavior of PSN near its dielectric peak. This work demonstrates the importance of H_{eff} methods that allow us to make a detailed investigation of the effects of nanoscale chemical ordering on dielectric properties.

The semiconductor industry is interested in HfO_2 as an alternate gate dielectric material. Experiments typically show significant numbers of defects in HfO_2. First principles calculations show that O vacancies are most stable on the 4-fold coordinated O site and that HfO_2 remains insulating with both a neutral and a 2+ charged vacancy. Work is underway to determine how each kind of vacancy affects the dielectric properties.

Quantitative modeling of mechanical behavior at the nanoscale requires connecting the large-scale elastic displacement fields experienced by the whole device to the small-scale atomistic regions where bond breaking can mark the initiation of device failure through fracture or plastic deformation. We are working to develop multiscale models for connecting the microscopic applied load in a nanoindentation experiment to the initiation of the first broken bond. The sample is a bulk Al single crystal with a <111> surface.

Contributors and Collaborators

S. Tinte, I. Levin (Ceramics Division, NIST); L. Levine, F. Terrazza (Metallurgy Division, NIST); A. Chaka (CSTL, NIST); A. van de Walle, M. Astar (Northwestern University); D.J. Singh (ORNL); W. Kleeman (University of Duisberg); U.V. Waghmare (J. Nehru Center for Advanced Scientific Research)

Advanced Materials for Energy Applications

Fundamental understanding, unique characterization facilities, and standardized materials and data form the technological basis for advances in materials for energy-related technologies. We are working to develop the metrology required to relate properties and performance of energy materials to processing/manufacturing routes via understanding the roles of chemistry, phase relations, and microstructure, to establish a sound physical basis for material system design.

Winnie Wong-Ng, Andrew J. Allen, Daniel A. Fischer, and Lawrence P. Cook

A challenge for the U.S. economy in the new millennium is for both emerging and mature industries to provide environmentally friendly, inexpensive, efficient, compact, and cutting-edge synergistic technologies for energy conversion, distribution, and storage applications. This project aims to facilitate commercialization of energy-related technologies by addressing various near-term and long-term materials issues.

The proliferation of portable telecommunication devices, computer equipment, and hybrid electric vehicles has created a substantial interest in manufacturing rechargeable Li-ion batteries that are less expensive, non-toxic, durable, and small in size and weight. The electronic structure of the electrode materials during the electrochemical cycling is particularly important to the implementation of Li-ion batteries. This year, we studied the electronic structure of the Li-ion deintercalated $Li_{(1-x)}Co_{1/3}Ni_{1/3}Mn_{1/3}O_2$ materials with soft X-ray absorption spectroscopy (XAS) at O *K*-edge and metal $L_{\alpha,\beta}$-edges, in combination with metal *K*-edge XAS spectra in the hard x-ray region to elucidate the charge compensation mechanism. We found that a large portion of the charge compensation during Li-ion deintercalation is achieved in the oxygen site due to the presence of Co.

Characterization of the triple phase boundary in solid oxide fuel cells (SOFC), where the electron- and ion-conducting phases and the gas transport (void) phases meet, is a priority in understanding SOFC performance and durability. By combining studies of the large microstructure scale range, accessible using the NIST-built ultrasmall-angle x-ray scattering (USAXS) facility at the Advanced Photon Source, with anomalous small-angle x-ray scattering (ASAXS) measurements, it has been demonstrated that ASAXS can provide the differential contrast for distinguishing between the ion- and electron-conducting phase morphologies.

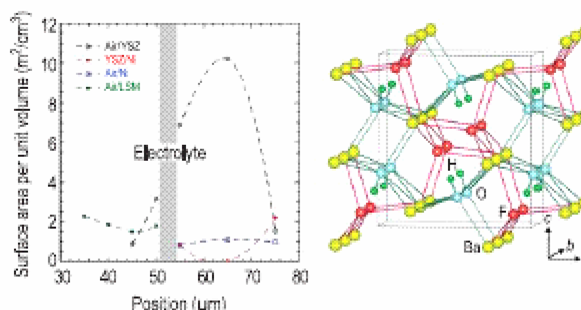


Figure 1: Spatial variation in component phase surface areas.

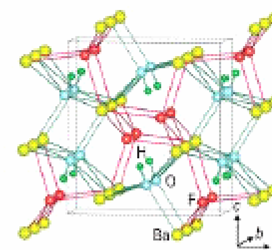


Figure 2: Structure of Ba(OH)F.

Figure 1 shows the spatial variation of the void/solid surface areas involving the ion-conducting yttria-stabilized zirconia (YSZ) and lanthanum strontium manganate (LSM), and Ni that are the electron-conducting phases in the cathode and anode layers.

Phase equilibria data are critical for the coated conductor high T_c materials for cable, generator, fly-wheel, and transformer applications. As an integral part of a DOE R&D program, phase diagrams were developed for the Ba-R-Cu-O (R= Tm and Yb) systems. The "BaF_2" process is a promising method for producing long-length coated conductors. We have investigated the role of low-temperature melt and intermediate superlattice phases in the formation of $Ba_2YCu_3O_{6+x}$ (Y-213). A new Ba(OH)F phase (Figure 2) was discovered that may be related to the low-temperature melting. The interaction of Y-213 with $SrTiO_3$ substrates was studied in terms of phase equilibria of the Ba-Sr-Y-Cu-Ti-O system.

In collaboration with the University of South Florida, we have characterized the structure and provided x-ray reference patterns for two clathrate phases ($Sr_8Ga_{16}Ge_{30}$ and $Cs_8Na_{16}Ge_{136}$) that are promising candidates for thermoelectric power conversion applications. The structure of $Na_{1-x}Ge_3$, which often coexists with $Cs_8Na_{16}Ge_{136}$, was also studied. Defining the industrial needs for thermoelectric metrologies and the uses of combinatorial approaches for materials optimization remain a high priority.

Contributors and Collaborators

F. Biancaniello, M. Green, R. Radebaugh, G. Nolas (USF); Z. Yang, I. Levin, Q. Huang, R. Feenstra (ORNL); A. Goyal, V. Maroni (ANL); W.S. Yoon (BNL); K.Y. Chung, X.Q. Yang, J. McBreen, M. Balasubramanian, C.P. Grey, J. Ilavsky (APS XOR); P.R. Jemian (University of Illinois); J. Ruud (GE), W. Dawson (Nex Tech); A. Virkar (University of Utah)

Thermochemical Metrology of Interfacial Stabilities

Hetero-interfaces are present in the majority of electronic materials and affect the operating characteristics of many devices. For successful fabrication and optimal performance of advanced designs, data on the behavior of interfaces during thermal processing is essential. Efficient measurement and prediction of the interfacial thermochemical stabilities of potentially useful materials combinations require new technology and new approaches, which are being developed in our laboratories.

Lawrence P. Cook, Mark Vaudin, and Martin L. Green

By using thin-film differential scanning calorimeters (DSCs), it is possible to study materials interactions on a very fine scale — both in terms of the mass of the samples involved, as well as the magnitude of the thermodynamic quantities measured. A further advantage of the thin-film DSC approach to thermochemical metrology is that it lends itself to combinatorial studies, in which compositionally graded thin films can be deposited over a DSC array. In this way, interfacial stabilities of various materials combinations can be rapidly evaluated. Our plan is to correlate thermochemical data from DSC arrays with characterization by x-ray microdiffraction, using an area detector and an automated stage to sample each element of the array. The products formed during interfacial reaction will be identified, and these experiments will form the basis for more detailed examination of the kinetics of reaction for appropriate materials combinations.

To date, our efforts on thin-film DSC have concentrated on proof-of-concept using well-known thermochemical events such as the melting of Sn (232 °C), the ε/α transition in metallic Co (422 °C), and the reaction between elemental Si and Ni thin films (kinetically determined). Studies of the latter two reactions are still in progress, but our preliminary studies of Sn melting have been completed. Figure 1 shows a 200 nm thick layer of Sn deposited on the sample side of a thin-film DSC sensor. The sample was deposited by pulsed laser deposition (PLD) through a 60 µm x 100 µm mask; a range of particle sizes is evident. Figure 2 is an uncalibrated average of multiple DSC scans from a sample like that in Figure 1. The melting (endothermic) and crystallization (exothermic) scans show multiple events, possibly related to differences in thermal behavior of different size fractions of the particles. A large thermal hysteresis between melting and crystallization is evident. This example suggests that the thin-film DSC is sensitive to variations in materials properties, in this case probably influenced by the interfacial tension between the metallic Sn in the core of the particles and the thin SnO_2 skin on the surface of the particles.

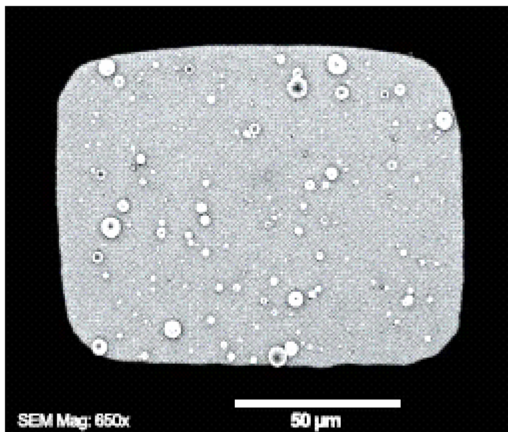


Figure 1: PLD-deposited Sn on DSC sensor.

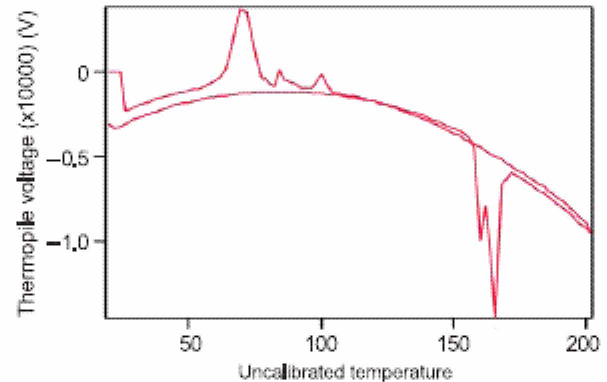


Figure 2: Thin-film DSC of Sn in Figure 1.

Work has already begun on a second generation of thin-film DSCs that will be optimized to give true nanometric sensitivity. This work is being done in collaboration with the University of Illinois at Urbana–Champaign. Currently, instrumentation is being set up to utilize the enhanced sensitivity that will be achieved through a chip design minimizing thermal mass. With the newer devices, near-adiabatic measurements will be possible.

Contributors and Collaborators

R. Cavicchi, C. Montgomery, S. Semancik, M. Carrier (Process Measurements Division, NIST); P. Schenck, W. Wong-Ng, J. Blendell (Ceramics Division, NIST); L. Allen (University of Illinois, Urbana–Champaign); M. Efremov (University of Wisconsin)

Materials for Advanced Si CMOS

Further size reduction of complementary-metal-oxide-silicon (CMOS) devices in the Si microelectronics industry, necessary for continued adherence to Moore's law, is currently materials limited. The gate stack, composed of the SiO_2 gate dielectric and the doped polycrystalline Si gate electrode, is no longer sufficient and must be replaced by new materials. Further, starting Si wafers must be strain engineered to increase carrier mobility and subsequent device performance. NIST plays an active role in both activities.

Martin L. Green

NIST/MSEL is poised to play important roles in the introduction of new materials to the Si microelectronics industry, so that further scaling (size reduction) may be enabled. One example is the advanced gate stack for Si CMOS. To enable further device scaling, the capacitive equivalent thickness (CET) of the gate stack must be 0.5 nm to 1.0 nm. This will not be achievable with existing SiO_2/polycrystalline Si gate stacks. Since replacements for SiO_2 have already been identified, it is now particularly important to replace the doped polycrystalline Si gate electrode with a true metal. Given the large number of possible choices of alloys for the metal gate electrode, the combinatorial approach is seen as the most effective way of identifying alloys possessing the proper work function and stability as metal gates on HfO_2.

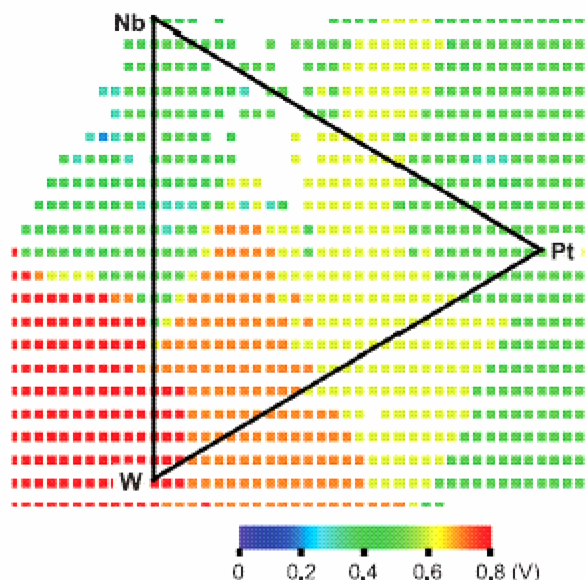


Figure 1: Flatband voltage measurements obtained from capacitance — voltage data, for the Nb-Pt-W metal gate system.

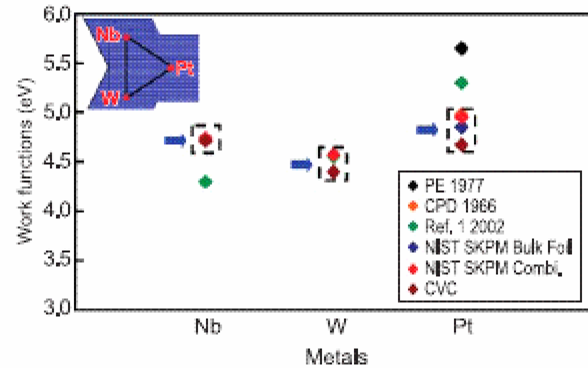


Figure 2: Comparison of work function data determined through combinatorial experiments and other techniques.

Figure 1 shows flatband voltage data, from which work functions may be derived, measured on hundreds of capacitors, where each small square represents a different metal gate composition in the Nb-W-Pt ternary system. Variations in flatband voltage, due to alloy composition, are readily observed. As can be seen from Figure 2, work functions determined through combinatorial experiments are in excellent agreement with those determined by other, less straightforward means.

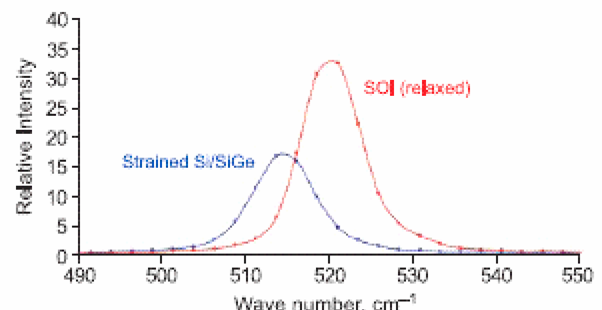


Figure 3: Raman shift of strained Si with respect to relaxed Si.

Another example of NIST activity in advanced Si materials is strained Si, which is increasingly being used to enhance carrier mobilities in high performance devices. Figure 3 shows the characteristic Raman shift that accompanies strain in the Si lattice. MSEL/NIST is working to calibrate the Raman shift to an absolute strain measurement (via precision lattice parameter determination) to facilitate the introduction of this technology.

Contributors and Collaborators

D. Black, K.-S. Chang, T. Chikyow (NIMS, Japan); M. Gardener (Sematech); D. Josell, R. Lei (Intel); P. Majhi (Sematech); A. Paul, P. Schenck, W. Wong-Ng, J. Suehle (EEEL/NIST); I. Takeuchi (University of Maryland)

Metrology and Standards for Electronic and Optoelectronic Materials

The electronics and optoelectronics industries require accurate materials properties data to improve their device fabrication, modeling, and evaluation processes. Our recent activities have emphasized optical and structural metrologies applicable to wide bandgap semiconductor nanowires, and UV Raman spectroscopy for the evaluation of strain in strained silicon-on-insulator structures.

Lawrence H. Robins, Igor Levin and Albert J. Paul

Nanowires, defined as semiconductor or metal structures having a quasi-cylindrical geometry with diameter in the range 1 to 100 nm, are expected to have a major impact on future electronic and optoelectronic technologies. A NIST program was recently started to develop growth and manipulation techniques, metrologies, and test structures for semiconductor nanowires, based on GaN growth by molecular beam epitaxy (MBE), and ZnO growth by the catalyst-assisted vapor-liquid-solid (VLS) method. We are contributing to this program by developing metrologies for structural and optical characterization of the nanowire samples and test structures.

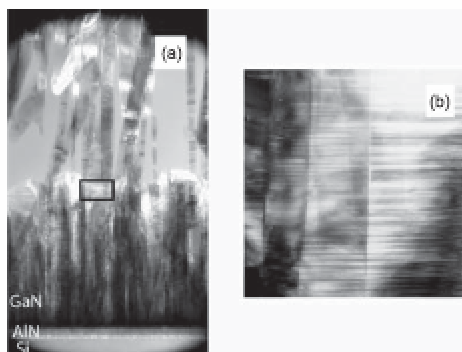


Figure 1: Dark-field TEM images of GaN nanowire and matrix structure grown on AlN/Si. (a) Lower magnification image shows nanowires and unwanted matrix layer both growing from AlN buffer. (b) Higher magnification image shows basal-plane defects in matrix layer.

TEM structure of one of our first GaN nanowire samples, grown by MBE on an AlN buffer layer on Si(111), is shown in Figure 1. The sample contains both nanowires and a rough, faceted "matrix" layer (lower half of Figure 1(a)). Growth of most nanowires appears to initiate at the AlN buffer, rather than within the matrix layer. The matrix layer contains a high density of basal-plane defects, which produce striations in the dark-field image (Figure 1(b)), and also give rise to streaking in electron diffraction (not shown here). In contrast, the nanowires appear free of extended defects in TEM.

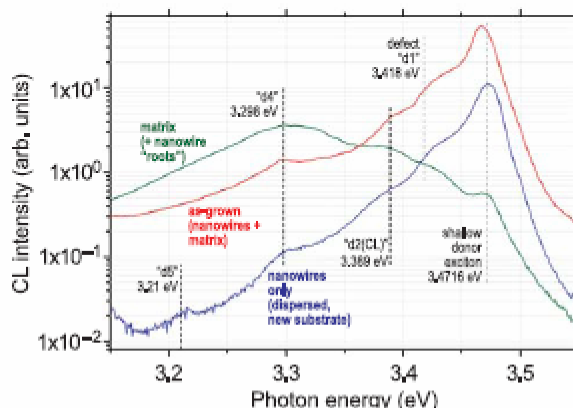


Figure 2: Low-temperature CL of GaN nanowire sample. Red curve: as-grown. Blue curve: nanowires only, removed from matrix layer. Green curve: matrix layer + nanowire "roots," with tops of nanowires polished off.

The nanowire structure from Figure 1 was characterized by low-temperature cathodoluminescence (CL), as shown in Figure 2. The CL of the nanowires is dominated by excitons bound to shallow donor impurities (3.4716 eV peak) while the matrix layer CL is dominated by excitons bound to structural defects (peaks "d1" through "d5") that may be related to the defects seen by TEM.

Similarly to the GaN samples, the ZnO samples were found to contain a highly defective "matrix" layer together with relatively defect-free vertical nanowires, but the matrix layer in the ZnO was found to consist of horizontally growing nanowires. The choice of metal catalyst (Ag, Au, or Cu-containing alloy) for VLS growth of the ZnO samples was found to have a strong affect on the CL spectra, ascribed to catalyst doping of the ZnO and concomitant formation of catalyst-related impurity levels. Our characterization results on GaN and ZnO nanowires were reported at the 2005 Electronic Materials Conference (Santa Barbara, CA, June 22–24).

Contributors and Collaborators

A. Davydov, A. Motayed (Metallurgy Division, NIST); J. Barker, K. Bertness, N. Sanford (Optoelectronics Division, NIST); B. Nikoobakht (Surface and Microanalysis Science Division, NIST); R.Z. Lei (Intel Corporation); G. Celler, M. Kennard (SOITEC)

Data and Data Delivery

The economic importance of technical data has grown steadily over the past three decades. NBS/NIST took an important lead early in the effort to emphasize the pervasive use and impact of reliable materials property data. As early as 1985, former NBS Director, E. Ambler, noted that the need for property data had become a "national priority," while the National Research Council repeatedly observed that there is a persistent "critical national need" for materials property data.

The NIST Materials Science and Engineering Laboratory (MSEL) has been a prominent leader in responding to this national need. By design, the scope of the MSEL effort is evolutionary and responds to the ever increasing advances in technology.

Crystallographic Databases *[Contact - V. Karen]*: This project encompasses the two most venerable efforts in MSEL, each of which recently has had a major new release. In collaboration with Fachinformationszentrum Karlsruhe (FIZ, Germany), a CDROM version of the FIZ/NIST Inorganic Crystal Structure Database (ICSD) has been released providing the full structural data, *i.e.*, lattice parameters and atomic coordinates, for more than 80,000 compounds.

Phase Equilibria and Properties of Dielectric Ceramics *[Contact - T. Vanderah]*: The long-standing collaboration between NIST and the American Ceramic Society (ACerS) has continued with the completion of a new topical volume in the NIST/ACerS Phase Equilibria Diagrams series and the release of version 3.1 of the CDROM product. An integrated theoretical and experimental effort is underway to predict and measure phase equilibria and electronic behavior in dielectric oxide ceramic systems. The work includes relaxor ferroelectrics, dielectrics for cellular infrastructure and hand-held devices, and dielectrics for low temperature co-fired ceramics for applications in multilayer ceramic integrated circuit technology.

Evaluated Materials Property Data *[Contact - R..Munro]*: All engineering designs of advanced materials require reliable data. Elasticity, strength, toughness, hardness, creep, expansion, conductivity, diffusivity, and durability are prominent among the data categories needed and desired for materials applications and development. The NIST effort is directed both towards the development of evaluated databases of these properties for structural and superconducting ceramics and towards the establishment of the data evaluation methodologies that form the foundation of reliable materials property data systems, and conducting assessments of data needs in emerging technologies.

Contact: Ronald G. Munro

Crystallographic and Phase Equlibria Databases

Crystallographic data models are used on a daily basis to visualize, explain and predict the behavior of chemicals and materials, as well as to establish the identity of unknown phases in crystalline materials. Phase diagrams are used throughout the ceramics industry to understand and control the complex phenomena which increasingly underlie advanced industrial material production and materials performance. Literally tens of thousands of structures and phase diagrams have been reported in the literature, all with varying degrees of reliability and completeness. This project develops, maintains, and disseminates comprehensive, critically-evaluated data in printed and in modern computerized formats, along with scientific software to exploit the content of these databases.

Vicky Lynn Karen, Terrell A. Vanderah, and Peter K. Schenck

The NIST Structural Database (NSD) and Inorganic Crystal Structure Database (ICSD) are comprehensive collections of crystal structures covering the literature from 1915. The ICSD database system now includes chemical and 3-dimensional structure information for more than 85,000 inorganic materials. The NSD database updates this year doubled the accessible data to more than 16,000 entries. These data and scientific software are licensed to instrument manufacturers, software vendors, and other third-party distributors as well as to individual researchers in industry and academia.

To meet the need for reliable phase diagram data, the NIST Phase Equilibria Data Center and the American Ceramic Society (ACerS) jointly publish a series of critically evaluated collections of phase diagrams. The series "Phase Equilibria Diagrams," originally published under the title "Phase Diagrams for Ceramists" (1964-1992), provides current, evaluated data on the phase equilibria of ceramics and related materials. The publications also provide bibliographic data, graphical representations, and analytical capabilities so that researchers have access to reliable, up-to-date data for use in designing, applying, analyzing, and selecting those materials. The published portion of the database includes more than 15,000 entries with over 20,000 phase diagrams contained in twenty-one books and a CD-ROM – over 53,000 units have been sold world-wide. Approximately 1000 new entries are collected from the primary literature each year.

Database products completed this year include *Volume XIV: Oxides*, edited by Robert S. Roth and Terrell A. Vanderah. This volume contains more than 1200 evaluated diagrams and more than 800 descriptive commentaries on oxide systems, primarily from literature published from the early 1980's through 2004. Volume XIV complements the earlier blue books on oxide systems (I-IV, VI, XI, XII, XIII), Annuals 91-93, and the topical publications on High T_c Superconductors, Zirconium and Zirconia Systems, and Electronic Ceramics I. Systems in this volume include a wide variety of metal, non-metal, and semi-metal oxides as well as numerous aluminates, silicates, and ferrites.

Also expected in 2005 is the release of version 3.1 of the CD-ROM Database (NIST Standard Reference Database Number 31), which will provide customers with comprehensive coverage of all diagrams and commentaries published to date, including Volume XIV.

Figure 1: Demonstration disk for Phase Equilibria Diagrams CD-ROM Database Version 3.1 (NIST Standard Reference Database Number 31), released in May 2005. This promotional item provides customers with a complete and searchable cumulative index for all published material. In addition, a small portion of the database (Annual 92) is included for customers to sample using the full functionality of the application.

Future database products include CD-ROM versions of selected books (High T_c Superconductors, Zirconium and Zirconia Systems, and Electronic Ceramics I), and a topical slice through the database, Phase Diagrams for Fuel Cells.

Contributors and Collaborators

The American Ceramic Society, R.S. Roth, M.A. Harne, C. Cedeno, J. You, N. Swanson, E. Farabaugh, K. Hill.

Fachinformationszentrum Karlsruhe, Germany; D. Watson (Cambridge Crystallographic Data Centre, UK); X. Li and A.Rajagopalan (Rensselaer, NY); A. Belsky (NIST Measurement Services Division).

Evaluated Materials Property Data

The internet has become the resource of first choice for technical materials information. The lack of publication constraints in this medium, however, poses a serious problem regarding the quality of the information retrieved from unqualified sources. The U. S. National Research Council, CODATA International, ISO (the International Organization for Standards), and ASTM International have separately indicated the urgent, timely, and persistent need for evaluated data and for standards, protocols, or tools by which the reliability of such information sources can be assessed.

Ronald Munro and Charles Sturrock

Representations of Data: Real-time simulations of materials behavior and product performance demand property data as a function of temperature and as a function of material characteristics such as porosity. Traditionally, such data have been available in tabulations of property values. Discrete compilations, however, can be cumbersome to use in automated computations, particularly if the computational algorithms and the data are on different computers. Furthermore, discrete compilations are subject to inconsistencies in value selection criteria when the operating point is not one of the discrete values in the discrete set. Analytic representations can resolve this problem and provide additional value-added assessments.

A study addressing this issue for elastic moduli data has been completed using an analytical model that provides a self-consistent representation of the simultaneous temperature and porosity dependence of the elastic and bulk moduli of polycrystalline ceramics. The data for 24 oxide ceramics were obtained from the Ceramics WebBook collection, NISTIR 6853, compiled previously in this project.

Data Evaluation Tool: The central theme developed in the NIST Recommended Practice Guide, SP960-11, is that the function of data evaluation is to ascertain the credibility of data. Based on the analysis given in that Guide, we have used hypertext markup language (HTML) to implement an interactive protocol according to which materials property data can be assessed and classified into seven different levels of acceptable data. This tool, accessible *via* the Ceramics WebBook (http://www.ceramics.nist.gov/IDELA/IDELA.htm), leads the user through a series of questions in the manner of a decision tree. Upon reaching a conclusion, the tool presents a summary of the decision sequence along with the determined data evaluation level.

Data and Informatics Needs in Biomaterials: Used in medical devices, biomaterials are natural or artificial materials that interact with biological systems to replace or augment the function of living tissue. In addition to nonviable medical implants, biomaterials are also found in tissue engineering and artificial organ applications where living cells are used.

With an estimated 13 % annual growth rate, biomedical devices and the materials from which they are made represent one of the fastest growing segments of both the biotechnology and materials industries. Approximately 8 % to 10 % of the U.S. population has a medical implant; examples include: intraocular lens, heart valve, pacemaker, coronary/vascular stent, breast implants, and joint prosthesis. Even more common biomaterials include contact lenses, dental amalgams, sutures, and edible coatings for drug tablets or capsules. Hence virtually every American at some point consumes biomaterials.

Biomaterials property data are widely scattered across the relevant literature and the internet. However, because few standards exist for biomaterial property measurement, these data often are not directly comparable. As a result, there are few biomaterials property databases: the only known online source documents properties of primarily dental materials; three other databases contain biomolecular structural data. The data from these printed and electronic sources are used to characterize biomaterials, to design biomedical devices, to screen or select materials for biomedical applications, to simulate biomaterial performance, to develop protocols for the synthesis of biological macromolecules, and to substantiate hypotheses and theory.

An assessment of the data and informatics needs in biomaterials is presently under development. The study will address the primary issues, needs, barriers, and opportunities surrounding biomaterials property data and also will examine the key players and their roles in biomaterials technology. Research highlights, resources, and data delivery mechanisms will serve to inform and guide future efforts in this burgeoning field.

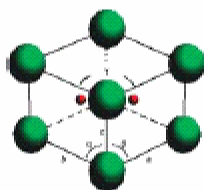
Contributors and Collaborators

Joyce Harris (Ceramics Division, NIST)

Data, Reference Materials, and Measurement Methods

The Ceramics Division has been a vigorous participant in support of NIST's core mission as a Measurement Standards Institute. The Division has made significant contributions to all aspects of standard reference databases, standard reference materials, and standard measurement methods and practices. As may be observed here, the products established by the Division provide a substantial resource spanning a considerable sector of materials metrology.

DATABASES



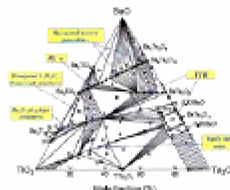
Crystallography

NIST principal investigator: V.L. Karen

NIST Standard Reference Database 15
NIST/Sandia/ICDD Electron Diffraction Database. Chemical, physical, and crystallographic information for more than 81,500 materials, including minerals, metals, intermetallics, and general inorganic compounds; the database and associated software enable highly selective identification procedures for microscopic and macroscopic crystalline materials. Available for purchase at **http://www.nist.gov/srd/nist15.htm**.

NIST Standard Reference Database 83
NIST Structural Database. Crystallographic and atomic position information for metallic crystalline substances, including alloys, intermetallics and minerals; the database is distributed in an ASCII format, convenient for reading into a variety of database management systems or processing by independent software routines. Available for purchase at **http://www.nist.gov/srd/nist83.htm**.

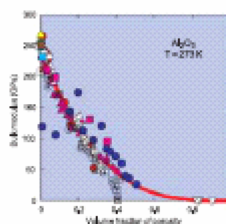
NIST Standard Reference Database 84
FIZ/NIST Inorganic Crystal Structure Database, Release 2004/1 (February 2004); produced cooperatively by the Fachinformationszentrum Karlsruhe (FIZ) and NIST; a comprehensive collection of full structural crystallography data of inorganic compounds; contains more than 70,000 entries. Available for purchase at **http://www.nist.gov/srd/nist84.htm**.



Phase Equilibria Diagrams

NIST principal investigator: T.A. Vanderah

NIST Standard Reference Database 31
NIST/ACerS Phase Equilibria Diagrams. Produced jointly by NIST and the American Ceramic Society; thirteen regular book volumes, four topical volumes, three annual volumes, and a computerized database on CD ROM; more than 53,000 units have been sold; the current CD contains approximately 20,000 critically evaluated diagrams and 15,000 expert commentaries. (Free demo CD available from NIST.) Available for purchase at **http://www.nist.gov/srd/nist31.htm**.



Materials Properties

NIST principal investigator: R.G. Munro

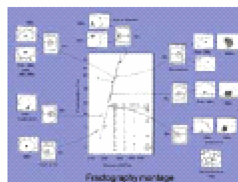
NIST Standard Reference Database 30
Structural Ceramics Database. Physical, mechanical, and thermal properties; more than 38,000 numeric values. Available online at **http://www.ceramics.nist.gov/srd/scd/scdquery.htm**.

NIST Standard Reference Database 62
High Temperature Superconductors. Physical, mechanical, thermal, and superconducting properties; more than 30,000 numeric values. Available online at **http://www.ceramics.nist.gov/srd/hts/htsquery.htm**.

NIST Property Data Summaries

Focused studies with comprehensive property sets for specific materials and topical studies focused on one property for a wide range of materials. Available as follows:

- Alumina, **http://www.ceramics.nist.gov/srd/summary/scdaos.htm**
- Silicon Carbide, **http://www.ceramics.nist.gov/srd/summary/scdscs.htm**
- Titanium Diboride, **http://www.ceramics.nist.gov/srd/summary/scdtib2.htm**
- Yttrium Barium Copper Oxide, **http://www.ceramics.nist.gov/srd/summary/htsy123.htm**
- Elastic Moduli Data, **http://www.ceramics.nist.gov/srd/summary/emodox00.htm**
- Fracture Toughness Data, **http://www.ceramics.nist.gov/srd/summary/ftmain.htm**
- Fracture Data for Oxide Glasses, **http://www.ceramics.nist.gov/srd/summary/glsmain.htm**



Fractography

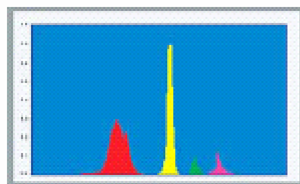
NIST principal investigator: G.D. Quinn

Characterization of Fracture Origins

Provides an efficient and consistent methodology to locate and characterize fracture origins in advanced ceramics. May be used in conjunction with ASTM standard C-1322. Available online at **http://www.ceramics.nist.gov/webbook/fracture/fracture.htm**.

STANDARD REFERENCE MATERIALS

SRMs produced by the Ceramics Division are available for purchase at **http://ts.nist.gov/ts/htdocs/230/232/232.htm**.



Particle Size Metrology SRMs

NIST principal investigator: J. Kelly

Standard Reference Material 1021

Glass Beads — Particle Size Distribution, a particle size standard for size range 2 µm to 12 µm.

Standard Reference Material 1003c

Glass Beads — Particle Size Distribution, a particle size standard for size range 20 µm to 50 µm.

Standard Reference Material 1004b

Glass Beads — Particle Size Distribution, a particle size standard for size range 40 µm to 150 µm.

Standard Reference Material 1017b

Glass Beads — Particle Size Distribution, a particle size standard for size range 100 µm to 400 µm.

Standard Reference Material 1018b

Glass Beads — Particle Size Distribution, a particle size standard for size range 220 µm to 750 µm.

Standard Reference Material 1019b

Glass Beads — Particle Size Distribution, a particle size standard for size range 750 µm to 2450 µm.

Standard Reference Material 659

Particle Size Distribution for Sedigraph Calibration, a particle size standard for size range 0.2 µm to 10 µm.

Standard Reference Material 8010

Sand for Sieve Analysis.

Standard Reference Material 1982

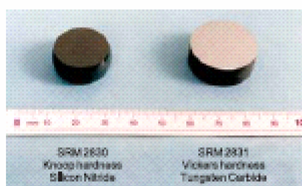
Zirconia Thermal Spray Powder — Particle Size Distribution, a particle size standard for size range 10 µm to 150 µm.

Standard Reference Material 1984

Thermal Spray Powder — Particle Size Distribution, Tungsten Carbide/Cobalt (Acicular), a particle size standard for size range 9 µm to 30 µm.

Standard Reference Material 1985

Thermal Spray Powder — Particle Size Distribution, Tungsten Carbide/Cobalt (Spheroidal), a particle size standard for size range 18 µm to 55 µm.



Mechanical Properties SRMs

NIST principal investigator: G.D. Quinn

Standard Reference Material 2830

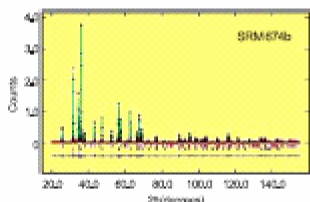
Knoop Hardness of Ceramics.

Standard Reference Material 2831

Vickers Hardness of Ceramics and Hardmetals.

Standard Reference Material 2100

Fracture Toughness of Ceramics.



X-Ray Metrology SRMs

NIST principal investigator: J. Cline

Standard Reference Material 640c

Silicon Powder Line Position/Profile SRM, silicon powder, used for calibration of line position and characterization of the instrument profile function, certified with respect to lattice parameter.

Standard Reference Material 660a

LaB_6 Powder Line Position/Profile SRM, LaB_6 powder, used to characterize the instrument profile function and calibrate line position, certified with respect to lattice parameter.

Standard Reference Material 675

Mica Powder Line Position (Low Angle) SRM, synthetic fluorophlogopite mica powder, used to characterize the instrument line position at low two-theta angle, certified with respect to lattice parameter.

Standard Reference Material 1976a

Instrument Response, a sintered alumina plate, certified with respect to lattice parameter and diffraction intensity as a function of two-theta angle (texture), used for general calibration of diffraction equipment, with respect to line position and intensity, via conventional data analysis methods.

Standard Reference Material 676

Alumina Powder for Quantitative Analysis, high purity alumina powder for general quantitative analyses via powder diffraction methods, certified with respect to lattice parameter.

Standard Reference Material 1878a

Quantification of Alpha Quartz, respirable (5 µm) powders, certified with respect to amorphous content, used primarily by the industrial hygiene community for quantification of quartz in airborne dust.

Standard Reference Material 1879a

Quantification of Cristobalite, respirable (5 µm) powders, certified with respect to amorphous content, used primarily by the industrial hygiene community for quantification of cristobalite in airborne dust.

Standard Reference Material 674b

Quantitative Analyses, four powders, Cr_2O_3, CeO_2, TiO_2 and ZnO, allows the user to match the linear attenuation of the standard to that of the unknown, certified for phase purity using neutron time-of-flight diffraction. Supplemental information will include the reference intensity ratio (RIR) or I/Ic value and the lattice parameters as determined with conventional x-ray diffraction.

Standard Reference Material 656

Si_3N_4 Powder for Quantitative Analysis, two samples of high purity silicon nitride powder, one high in the alpha phase while the other is high in the beta phase, for quantitative analyses via powder diffraction methods, certified with respect to phase purity and the alpha to beta phase ratio.

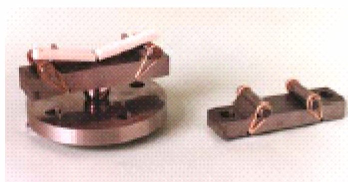
Standard Reference Material 2910

Calcium Hydroxyapatite, calcium hydroxyapatite powder for use in evaluating calcium apatites, primarily in the field of biological research, certified with respect to lattice parameters and phase purity.

Standard Reference Material 1979

Crystallite Size/Line Broadening, CeO_2 and ZnO powder which exhibits diffraction line profile broadening due to crystallite size effects, certified with respect to particle size via XRD line profile analysis, applicable to a range of materials research and industrial interests concerned with crystallite size determination via powder diffraction techniques.

STANDARD TEST METHODS



Mechanical Property Test Methods

NIST principal investigator: G.D. Quinn

ASTM C 1161 (2002)
Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature.

ASTM C 1322 (2002)
Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics.

ASTM C 1326 (2003)
Standard Practice for Knoop Hardness of Advanced Ceramics.

ASTM C 1327 (2003)
Standard Practice for Vickers Hardness of Advanced Ceramics.

ASTM F 2094 (2001)
Standard Specification for Silicon Nitride Bearing Balls. This standard addresses the basic quality, physical and mechanical properties, and test requirements for silicon nitride balls used for ball bearings and other specialty applications.

ASTM C 1211 (2002)
Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature.

ISO Standard 14704 (2000)
Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) — Test Method for Flexural Strength of Monolithic Ceramics at Room Temperature.

ISO Standard 18756 (2003)
Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) — Determination of Fracture Toughness of Monolithic Ceramics at Room Temperature by the Surface Crack in Flexure (SCF) Method.

ISO Standard 17565 (2004)
Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) — Test Method for Flexural Strength of Monolithic Ceramics at Elevated Temperature.

Ceramics Division FY05 Annual Report Publication List

Aguirre-Tostado, F. S., Herrera-Gomez, A., Woicik, J. C., Droopad, R., Yu, Z., Schlom, D., Karapetrova, E., Zschack, P., Pianetta, P., and Hellberg, S., "Elastic Anomaly for $SrTiO_3$ Thin Films on Si(001)," *Physical Review B,* **70**, *Rapid Communications*, 201403 (2004).

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Allen, A.J., Dobbins, T.A., Ilavsky, J., Zhao, F., Virkar, A., Almer, J., and DeCarlo, F., "Characterization of Solid Oxide Fuel Cell Layers by Computed X-ray Microtomography and Small-Angle Scattering," *Ceramic Engineering and Science Proc.*, **25** [3], pp. 275-280 (2004).

Andruzzi, L., Hexemer, A., Li, X., Ober, C. K., Kramer, E. J., Galli, G., Chiellini, E., and Fischer, D. A., "Control of Surface Properties Using Fluorinated Polymer Brushes Produced by Surface-Initiated Controlled Radical Polymerization," *Langmuir*, **20**, pp. 10498-10506 (2004).

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Bhushan, B., Liu, H., and Hsu, S. M., "Adhesion and Friction studies of silicon and hydrophobic and low friction films and investigation of scale effects," *ASME J. of Tribology*, **126**, pp. 583-590 (2004).

Black, D., "Using X-ray Topography to Inspect Surfaces of Single-Crystal Components," *International Journal of Applied Ceramic Technology*, **2** [4], pp. 336-343 (2005).

Bubeck, R. A., Dvornic, P. R., Hu, J., Hexemer, A., Li, X., Keinath, S. E., and Fischer, D. A., "Near Edge X-ray Absorption Fine Structure (NEXAFS) Studies of Copper Ion-Containing PAMAMOS Dendrimer Networks," *Macromol. Chem. Phys.*, **206**, pp. 1146-1153 (2005).

Burnett, D., Capitano, A., Gabelnick, A., Marsh, A., Fischer, D., and Gland, J., "In-situ Soft X-ray Studies of CO Oxidation on the Pt(111) Surface," *Surface Science*, **564**, pp. 29-37 (2004).

Burnett, D. J., Fischer, D. A., and Gland, J. L., "Soft X-ray Studies of Toluene Catalytic Oxidation on the Pt(111) Surface," *Journal of Physical Chemistry*, **108**, pp. 605-611 (2004).

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Ceramics Division

Chief
Debra L. Kaiser
Phone: 301-975-6119
E-mail: debra.kaiser@nist.gov

Deputy Chief
Ronald G. Munro
Phone: 301-975-6127
E-mail: ronald.munro@nist.gov

Group Leaders

Characterization Methods
Daniel A. Fischer
Phone: 631-344-5177
E-mail: daniel.fischer@nist.gov

Data and Standards Technology
Ronald G. Munro
Phone: 301-975-6127
E-mail: ronald.munro@nist.gov

Electronic and Optoelectronic Materials
Martin L. Green
Phone: 301-975-8496
E-mail: martin.green@nist.gov

Nanomechanical Properties
Grady S. White, Acting
Phone: 301-975-5752
E-mail: grady.white@nist.gov

Research Staff

Allen, Andrew
andrew.allen@nist.gov
Small-angle x-ray scattering
Small-angle neutron scattering
Ceramic microstructural analysis

Black, David
david.black@nist.gov
Defect microstructures
X-ray imaging

Blendell, John
john.blendell@nist.gov
Ceramic processing
Sintering and diffusion-controlled processes

Burdette, Harold
harold.burdette@nist.gov
X-ray optics
X-ray diffraction imaging
Instrumentation

Burton, Benjamin
benjamin.burton@nist.gov
Calculated phase diagrams
Dielectric ceramics

Cline, James
james.cline@nist.gov
Standard reference materials
High-temperature x-ray diffraction
Microstructural effects in x-ray diffraction
Rietveld refinement of x-ray diffraction data

Cockayne, Eric
eric.cockayne@nist.gov
First-principles methods
Phase equilibria and properties of dielectrics

Cook, Lawrence
lawrence.cook@nist.gov
High-temperature chemistry
Phase equilibria

Fischer, Daniel
daniel.fischer@nist.gov
Soft x-ray absorption fine structure
X-ray scattering
Surface science

Fuller, Edwin
edwin.fuller@nist.gov
Microstructural modeling and simulation
Microstructure effects on physical properties
Nanomechanical testing and properties
Toughening mechanisms

Gates, Richard
richard.gates@nist.gov
Tribo-chemistry of ceramics
Chemical analysis of ceramics
Nanometer film thickness measurements

Green, Martin
martin.green@nist.gov
Si microelectronic materials
Electronic ceramics
Combinatorial materials science

Hackley, Vincent
vince.hackley@nist.gov
Particle characterization
Particle dispersion and interface chemistry
Visco-elastic properties of suspensions

Harris, Joyce
joyce.harris@nist.gov
Data acquisition
Digitization and data entry

Hockey, Bernard
bernard.hockey@nist.gov
Transmission electron microscopy
Nanoindentation
Microstructural and defect characterization
Structure and composition of interfaces

Hsu, Stephen
stephen.hsu@nist.gov
Nanotribology
Nanolubrication
Magnetic hard disk interface
MEMs stiction

Kaiser, Debra
debra.kaiser@nist.gov
Electronic ceramics
Combinatorial material analysis

Karen, Vicky
vicky.karen@nist.gov
Crystallographic databases
Materials informatics
Lattice theory
Structural analysis

Kelly, James
james.kelly@nist.gov
Quantitative scanning electron microscopy
Image analysis
Microstructural analysis
Powder standards

Research Staff

Levin, Igor
- **igor.levin@nist.gov**
- Phase transitions in electronic ceramics
- Crystal structure determination
- Electron microscopy

Lum, Lin-Sien
- **lin-sien.lum@nist.gov**
- Particle characterization
- Instrumentation

Munro, Ronald
- **ronald.munro@nist.gov**
- Materials properties of advanced ceramics
- Data evaluation and validation
- Analysis of data relations

Nosonovsky, Michael
- **michael.nosonovsky@nist.gov**
- Nanotribology
- Nanomechanics

Paul, Albert
- **albert.paul@nist.gov**
- Laser physics
- Residual stress measurement

Quinn, George
- **george.quinn@nist.gov**
- Mechanical property test standards
- Standard reference materials
- Fractography

Robins, Lawrence
- **lawrence.robins@nist.gov**
- Defect identification and distribution
- Cathodoluminescence imaging/spectroscopy
- Photoluminescence spectroscopy
- Raman spectroscopy

Schenck, Peter
- **peter.schenck@nist.gov**
- Emission and laser spectroscopy
- Thin film deposition
- Computer graphics and image analysis
- Combinatorial materials analysis

Smith, Douglas
- **douglas.smith@nist.gov**
- Nanomechanics
- Instrumented indentation
- Surfaces forces
- Adhesion

Sturrock, Charles
- **charles.sturrock@nist.gov**
- Materials informatics
- Adaptive learning methods
- Predictive data mining

Vanderah, Terrell
- **terrell.vanderah@nist.gov**
- Solid-state chemistry
- Phase equilibria of microwave dielectrics

Vaudin, Mark
- **mark.vaudin@nist.gov**
- Electron microscopy
- X-ray and electron diffraction
- Modeling of grain-boundary phenomena
- Dielectric films

Wagner, Richard
- **richard.wagner@nist.gov**
- Nanoindentation
- Modeling of phase transformation
- Atomistic modeling of nanomechanics

White, Grady
- **grady.white@nist.gov**
- Residual stress measurements
- Mechanical reliability
- Raman spectroscopy

Windover, Donald
- **donald.windover@nist.gov**
- High-resolution x-ray diffraction
- X-ray reflectometry
- Standard reference materials

Woicik, Joseph
- **joseph.woicik@nist.gov**
- UV photoemission
- X-ray standing waves
- Surface and interface science

Wong-Ng, Winnie
- **winnie.wong-ng@nist.gov**
- X-ray crystallography and reference patterns
- Phase equilibria/crystal chemistry
- High temperature superconductors

Zurbuchen, Mark
- **mark.zurbuchen@nist.gov**
- Transmission electron microscopy
- Epitaxial growth
- Electronic ceramics

Research Associates

Cedeno, Christina
American Ceramic Society

Chang, K.-S.
University of Maryland

Chuang. T.
Nuclear Regulatory Commission

Cook, Robert
University of Maryland

Dapkunas, Stanley
Consultant

Farabaugh, Edward
American Ceramic Society

Fu, Z.
SUNY

Grobelny, J.
University of LODZ

Harne, Mary
Consultant

Hastie, John
Consultant

Henins, Albert
Consultant

Hill, Kimberly
Consultant

Ives, L.
Consultant

Jang, Mi-Hye
Yonsei University

Karlin, B.
Brookhaven National Laboratory

Kim, D. I.
Korean Institute of Science and Technology

Krayzman, V.
Consultant

Li, J.
University of Maryland

Li, Xiang
George Mason University

Liang, Yanang
Universided Oviedo, Spain

Long, Gabrielle
Argonne National Laboratory

Lowhom, N.
Clemson University

Mighell, Alan
Consultant

Namboodiri, P.
Leiden University

Otani, M.
Kanagawa Academy of Science and Technology

Patri, A.
National Cancer Institute

Price, W.
APWP/KT Consultants

Rajagopalan, Arun
George Mason University

Reitsma, M.
University of New Castle

Ritter, Joseph
Consultant

Roth, Robert
Viper Group

Sambasivan, S.
Brookhaven National Laboratory

Slutsker, J.
University of Maryland

Sparenberg, B.
University of Maryland

Stan, G.
KT Consultants

Swanson, Nils
American Ceramic Society

Yang, Seung Ho
Korea Advanced Institute of Science and Technology

Yang, Z.
Yunnan Normal University

Research Staff

You, Jim
American Ceramic Society

Zhang, H.
University of Maryland