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