Materials Structure Characterization

Fundamental to the field of materials science and engineering is the study of the relationships between processing, structure, properties, and performance of materials. Therefore, tools and techniques for the characterization of materials structure is a cornerstone of the field. The NIST Materials Science and Engineering Laboratory (MSEL) has a long tradition of supporting and developing measurement methods and facilities for materials structure characterization. Facilities within MSEL include optical and electron microscopy, optical and electron scattering and diffraction, a state-of-the-art x-ray diffractometer, the NIST Center for Neutron Research, and experimental stations at the National Synchrotron Light Source (NSLS) at Brookhaven Laboratory and at the Advanced Photon Source (APS) at Argonne National Laboratory. At the NSLS, NIST operates a soft x-ray station in partnership with Dow and Brookhaven National Laboratory. At the APS, NIST is a partner with the University of Illinois at Urbana/Champaign, Oak Ridge National Laboratory, and UOP in a collaboration called UNICAT. At both facilities, NIST scientists and researchers from industry, universities, and government laboratories perform state-of-the-art measurements on a wide range of advanced materials. NIST scientists have consistently advanced the limits of these facilities in order to improve spatial resolution and sensitivity needed, for example, to interrogate the microstructure of both highly anisotropic and/or gradient materials such as advanced thermal coatings and fuel cell systems. Studies currently underway at these facilities include: *in situ* measurements of nanoparticle production; structure and dispersion of carbon nanotubes; three-dimensional imaging of natural and artificial tissues; surface and subsurface damage in UV lithography optics; strain-induced ferroelectric transitions in thin films; and determination of molecular orientation and bond concentration on chemically heterogeneous surfaces.

The materials characterization program has a strong emphasis on electron microscopy. The MSEL Electron Microscopy Facility provides structure and compositional characterization of a wide range of materials. The facility consists of two transmission electron microscopes (TEMs), three scanning electron microscopes (SEMs), a specimen preparation laboratory, and an image analysis/computational laboratory. The JEM3010 TEM resolves the atomic structure and employs an energy selecting imaging filter and x-ray detector (EDS) for analytical characterization of thin foil specimens. The JSM6400 SEM employs electron backscattered diffraction/phase identification and EDS systems to characterize the texture and composition of materials. Highlights from the facility for FY2003 include: the computer domain is now active, providing streamlined user access and network file storage; a research collaboration with the NIST Semiconductor Electronics Division is underway to characterize quantum effects in confined Si devices; the size and shape of III–V quantum dots are characterized with the NIST Optoelectronics Division; and composition maps of electrodeposited nanowires with tunable magnetic properties are generated in collaboration with Johns Hopkins University.

This MSEL program also incorporates standards activities. A state-of-the-art x-ray diffractometer has been developed to study the metrology of powder diffraction in order to develop the next generation of diffraction standard reference materials. A variety of standard reference materials (SRMs) needed by the U.S. polymers industry, research laboratories, and other federal agencies have recently been developed: polyethylene of narrow mass distribution; nonlinear fluids for rheological measurements; melt flow standards; and the first reference biomaterial, an orthopedic grade ultra-high molecular weight polyethylene.

Recent program activities utilize matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MS) to address the need for improved characterization of the molecular structure of polyolefins, a dominant commercial polymer. This work has extended the upper mass limit detectable by MS to 15,000 g/mol and, by observing individual oligomeric species by mass, has revealed details of the molecular structure.

A major cross-cutting activity within this program is the multi-scale, multi-modal imaging and visualization project. The goal of this project is to combine disparate sets of 3-D imaging data that contain complementary information on overlapping length scales to produce an interactive visualization scheme for multivariate data sets. This year focused on further improvements and additions to the suite of imaging tools for tissue engineering metrology. Osteoblasts cultured in a poly (ε-caprolactone) scaffold have been imaged, demonstrating the advantages of optical coherence and confocal fluorescence microscopies over conventional laser scanning confocal microscopy. This instrument was upgraded by completely rebuilding the image acquisition software, hardware, and optical train. These improvements enabled an increase in image acquisition speed (2X) and accuracy (from 5 μm to 360 nm).

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