Materials Structure Characterization

Materials science and engineering is the area of science concerned with understanding relationships between the composition, structure, and properties of materials and the application of this knowledge to the design and fabrication of products with a desired set of properties. Thus, measurement methods for the characterization of materials structure are a cornerstone of this field. MSEL supports a wide array of techniques and instrumentation for materials measurements. Facilities include optical and electron microscopy, optical and electron scattering and diffraction, and major x-ray facilities at the National Synchrotron Light Source (NSLS) at Brookhaven Laboratory, and at the Advanced Photon Source (APS) at Argonne National Laboratory.

Synchrotron radiation sources provide intense beams of x-rays enabling leading-edge research in a broad range of scientific disciplines. Materials characterization using x-rays from synchrotron sources forms a major part of the Materials Structure Characterization Program. This includes the development and operation of experimental stations at the NSLS and at the APS. 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. Studies currently underway include: ceramic coatings; defect structures arising during deformation of metals, ceramics, and polymers; defect structures in semiconductors and single-crystal proteins; and atomic-scale and molecular-scale structures at surfaces and interfaces in polymeric, catalytic, and metal/semiconductor systems.

Ceramic powders are precursors for over 80% of ceramic manufacturing. As a result, a major focus in the Ceramics Division is the accurate and reliable measurement of the physical and chemical properties of ceramic powders, including sub-micrometer and nanometer sized powders. These measurements are critical to ensuring processes and products of high quality, minimal defects, and consequent economic benefits. Another area of concern to ceramic manufacturing is powder dispersion in a fluid vehicle for shape forming and other uses. The chemical and physical characteristics of powders dispersed in liquids are evaluated to understand the influence of surface charge, dissolution, precipitation, adsorption, and other physicochemical processes on the dispersion behavior. In addition to these activities, standard reference materials for use as primary calibration standards and national/international standards for particle size and size distribution, pore volume, and particle dispersion measurements are being developed in collaboration with industrial partners, measurement laboratories, and academic institutions in the U.S., Europe, and Asia.

The NIST effort in materials characterization has a strong emphasis on electron microscopy, which is capable of revealing microstructures within modern nanoscale materials and atomic-resolution imaging and compositional mapping of complex crystal phases with novel electronic properties. The MSEL microscopy facility consists of two high-resolution transmission electron microscopes (TEM) and a high-resolution field-emission scanning electron microscope (FE-SEM) capable of resolving features down to 1.5 nm. Novel experimental techniques using these instruments have been developed to study multilayer and nanometer-scale materials.

Through this MSEL Program, measurement methods, data, and standard reference materials (SRMs) needed by the U.S. polymers industry, research laboratories, and other federal agencies are provided to characterize the rheological and mechanical properties of polymers and to improve polymer processibility. In response to critical industry needs for *in-situ* measurement methodologies, a substantial effort is underway to develop optical, dielectric, and ultrasonic probes for characterizing polymer processing. Improved methods for determining molecular mass distribution of polymers are developed because of the dramatic effect it has on processibility and properties. Mechanical properties and performance are significantly affected by the solid-state structure formed during processing. Importantly, unlike many other common engineering materials, polymers exhibit mechanical properties with time dependent viscoelastic behaviors. As a result, techniques are being developed that measure the solid-state structure and rheological behavior of polymeric materials. Recent program activities exploit advances in mass spectrometry using matrix assisted laser desorption ionization (MALDI) to develop a primary tool for the determination of the molecular masses of synthetic polymers, with particular emphasis on commercially important polyolefins. The polymer industry and standards organizations assist in the identification of current needs for SRMs, and based on these needs, research on characterization methods and measurements is conducted leading to the certification of SRMs.

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