Nanoscale Characterization of Materials

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Guest Editors

One of the dominant trends in current research in materials science and related fields is the fabrication, characterization, and application of materials and device structures whose characteristic feature sizes are at or near the nanometer scale. Achieving an understanding of—and ultimately control over—the properties and behavior of a wide range of materials at the nanometer scale has therefore become a major theme in materials research. As our ability to synthesize materials and fabricate structures in this size regime improves, effective characterization of materials at the nanometer scale will continue to increase in importance.

Central to this activity are the development and application of effective experimental techniques for performing characterization of structural, electronic, magnetic, optical, and other properties of materials with nanometer-scale spatial resolution. Two classes of experimental methods have proven to be particularly effective: scanning-probe techniques and electron microscopy. In this issue of *MRS Bulletin,* we have included eight articles that illustrate the elucidation of various aspects of nanometer-scale material properties using advanced scanning-probe or electron-microscopy techniques. Because the range of both experimental techniques and applications is extremely broad—and rapidly increasing—our intent is to provide several examples rather than a comprehensive treatment of this extremely active and rapidly growing field of research.

Four of the articles in this issue focus primarily on nanoscale materials characterization using scanning-probe techniques. Since its invention in the early 1980s, scanning tunneling microscopy (STM) and techniques such as atomic force microscopy that have evolved from it—to which we refer collectively as scanning-probe microscopy—have become tools of paramount importance in fundamental studies of surfaces. Furthermore, rapid progress in the development of new scanning-probe techniques and in the commercial availability of scanning-probe instrumentation has led to increasingly widespread application of these techniques to problems of both scientific and technological significance. Their growing importance is a natural consequence of the desire to design and fabricate ever smaller structures to improve performance or achieve new functionality in materials and devices, and of the limitations of more traditional experimental techniques in performing detailed and comprehensive characterization at the nanometer to atomic scale.

An example of the extension of STM from fundamental surface studies to atomic-scale characterization of semiconductor heterostructures and devices is provided by Edward T. Yu in his article on cross-sectional STM studies of III-V semiconductor heterostructures. In this work, tunneling measurements performed on cross sections of epitaxial layers exposed by cleaving under ultrahigh vacuum provide detailed information about the atomic-scale structure of heterojunction interfaces and alloy layers. Correlation of information obtained from cross-sectional STM studies with epitaxial-growth conditions, complementary characterization studies, and device behavior then provides a comprehensive picture of the relationships among phenomena occurring during epitaxial growth, the resulting atomic-scale morphology and electronic properties of heterostructures, and various aspects of device performance.

Optical characterization of materials with spatial resolution in the sub-100 nm regime can be performed by combining optical measurements with scanning-probe technology, as exemplified in the near-field scanning optical microscopy (NSOM) studies described by J.W.P. Hsu. Her article discusses the application of NSOM to the nanoscale characterization of defect structures in a variety of materials, with particular emphasis on the characterization of individual defects and the correlation of the optical and electronic activity associated with specific defects with structural features observable in topographic images. She presents as specific examples studies of strain-relaxed $Si_{1-x}Ge_x$ films and of perovskite oxide crystals and thin films, and also gives an overview of the application of NSOM to the characterization of optical and optoelectronic properties in a broad range of materials and device structures.

R. Wiesendanger provides a description of the correlation among nanometer-scale structural, electronic, and magnetic properties of thin magnetic films as elucidated in studies of ultrathin magnetic films using STM and scanning tunneling spectroscopy, spin-polarized scanning tunneling spectroscopy, and magnetic-force microscopy. Scanning tunneling spectroscopy, in which the tunneling current is measured as a function of voltage, allows atomic-scale structure and local electronic properties to be correlated and, for Fe deposited on W(110), provides a method to distinguish chemically between Fe and W atoms through variations in local conductivity. Spin-polarized scanning tunneling spectroscopy, in which tunneling measurements are performed using a magnetic-probe tip, offers the possibility of probing the electronic, magnetic, and structural characteristics of materials with nanometer-scale resolution. Ultrahigh-vacuum magnetic-force-microscopy studies of Co films on Si(100) are also discussed.

The work described thus far focuses exclusively on the characterization of various solid materials with nanometer-scale resolution. In the fourth article, Miquel Salmeron and co-workers discuss the application of a technique they have dubbed scanning polarization force microscopy to the study of liquid surfaces and films. The use of this technique to investigate the structure of thin liquid films and droplets, condensation phenomena, and corrosion processes is described. These investigations are providing new insights into the atomic-scale origin and nature of macroscopic concepts such as wetting, spreading, and contact angle, and illustrate the potential of scanning-probe techniques for investigation of nanoscale dielectric properties of liquids and molecules, as well as clarification of the detailed nature of a wide range of liquid-solid interactions.

Highly complementary to the surface view provided by scanning-probe techniques, transmission electron microscopy offers the ability to characterize buried interfaces in a wide range of materials. Their crystallographic, compositional, and electronic structures can be investigated at the nanoscale level and in many cases even at the atomic scale. Detailed characterization of interface structure and composition, and its evolution with time and temperature, is of profound fundamental and technological importance. The performance of a large range of modern materials and devices either is limited by internal interfaces or is reliant on them for the desired characteristics. Dramatic effects of grain boundaries are often seen in the electronic properties, such as reduced carrier densities, lifetimes, and mobilities in semiconductors; reduced efficiency of detectors and flat-panel displays; and reduced critical currents in superconductors. Conversely electroceramics such as varistors, capacitors, and sensors rely on the effects of grain boundaries for their operation. In general a fundamental understanding of any of these phenomena has yet to be achieved; advanced imaging techniques, assisted in many cases by state-of-the-art theoretical modeling, can begin to unravel structure-property relationships in these technologically important materials.

In the field of metal-ceramic composites, M. Wagner and co-workers describe high-resolution transmission and analytical electron microscopy investigations of several metal-ceramic "model" systems that illustrate the behavior of various types of metal-ceramic interfaces. The need to understand the nature, at the atomic scale, of metal-ceramic interfaces has driven the investigation of a variety of relatively simple "model systems." Quantitative comparison of electron-microscopy results with theoretical, atomic-scale models of these interfaces, combined with additional characterization of thin-film growth using STM, provides fundamental insights into the nature of bond formation at metal-ceramic interfaces and consequently a sound basis from which to guide the development of more complex material systems required in most technological applications.

U. Dahmen and co-workers discuss the equilibrium shapes of nanoscale inclusions in metals, with a view to understanding how to tailor the optical, magnetic, or structural properties of nanophase materials through control of grain size and shape. Using high-resolution electron microscopy, they find that the equilibrium shape of small inclusions is dependent on their size and in particular that certain "magic sizes" are preferred over others. The preferred sizes are those with minimum residual strain energy. Inclusions on grain boundaries show more complex shapes, which are dependent on boundary orientation.

M.F. Chisholm and S.J. Pennycook discuss the atomic structure of grain boundaries in silicon, showing how they are constructed from well-defined sequences of regular lattice-dislocation cores. Using Z-contrast imaging, the individual cores are seen to have the structure predicted long ago by Hornstra, though the actual sequences differ from previously proposed models as they include many redundant dislocations, in the form of pairs of dislocations of equal and opposite Burgers vectors. The observation that boundaries follow a well-defined sequence of known dislocation core structures, even when placed in a continuous sequence, suggests that more general grain boundaries could also be constructed using the same core structures.

Finally N. Tanaka and T. Kizuka show spectacular advances in the imaging of dynamic processes, combining atomic spatial resolution with high temporal resolution. Tungsten atoms are shown diffusing over the (100) surface of MgO, and are seen to occupy both Mg and O sites. New insights into the mechanisms of grain-boundary migration, metal-mediated crystallization, and tip/surface interactions in atomic force microscopy are revealed. Subnanometer electron-beam lithography is also demonstrated with the drilling of holes in MgO having both subnanometer size and subnanometer separation; in addition the ability to investigate damage mechanisms *in situ* is illustrated.

Together the articles in this issue of the *Bulletin* provide a sampling of the new and often unique insights arising from the nanoscale characterization of materials, through both scanning-probe and advanced electron microscopy techniques. Characterization techniques—both existing and new—that offer the ability to elucidate material properties with nanometer-scale spatial resolution will play a central role in much of the progress to be made in nanometer-scale science and technology. Materials research in this area is growing rapidly and promises to provide fertile ground for exploration and innovation in the coming years. □

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