

## Nanomechanics and Standards

*Although much of the focus in nanotechnology is on the electronic and magnetic properties of nanoscale devices, there are many applications where device performance depends on the mechanical properties of the materials used. Stress and interfacial adhesion affect the reliability of multilayer electronic structures. Devices with micro- and nano-scale moving parts, such as cantilevers, gears and micro-mirror arrays, suffer mechanical fatigue and premature failure for reasons that are often not well understood. We develop test methods and standards for measuring mechanical properties at the nanoscale.*

### Douglas T. Smith

In the world of macroscopic mechanical testing, many well-characterized, standardized test methods are available to measure elastic modulus, hardness, strength, fracture toughness, adhesion, residual stress and a host of other properties critical to the successful design of reliable macroscopic structures. Often, knowledge of these same properties is just as critical to the design of micro- and nano-scale devices, but in this realm, accurate, reliable test methods for determining mechanical properties are rare. We are working on developing and evaluating techniques for mechanical property measurement, helping to draft standard test methods and developing Standard Reference Materials (SRMs) and force calibration methodology for low-force (mN down to nN) mechanical testing.

One of the most commonly used techniques for determining hardness and elastic modulus of small volumes of materials is instrumented indentation testing (IIT), often referred to as nanoindentation, in which a diamond indenter is pushed into a specimen surface and the force on, and displacement into, the surface are recorded, and those data are analyzed. The technique is capable of providing information on the elastic and plastic deformation of a specimen for indentations as shallow as 10 nm to 20 nm, and it is routinely used to measure the mechanical properties of thin films. However, until now there have been no accepted test methods that would allow IIT to be used as part of a thin-film or coating product specification. We are working with both the American Society for Testing of Materials, ASTM (E28.06.11), and the International Standards Organization, ISO (TC 164/SC 3), on draft standards for IIT; an ISO document for the technique is now at the level of Final Draft International Standard (FDIS 14577). In addition, the Ceramics and Materials

Reliability Divisions at NIST are working with the Bundesanstalt für Materialforschung und–prüfung (BAM) in Germany to develop joint thin-film SRMs (CRMs, or Certified Reference Materials, in Europe) for use in IIT machine verification. This effort involves the comparison of IIT results to measurements done in the Materials Reliability Division using surface acoustic wave spectroscopy. Results on SiO<sub>2</sub> and TiO<sub>2</sub> coatings show the techniques to be in good agreement.

Other intercomparisons of thin-film mechanical test methods are underway in Technical Working Area (TWA) 22 of VAMAS (Versailles Project on Advanced Materials and Standards, [www.vamas.org](http://www.vamas.org)). Here, international round robin testing for hardness, elastic properties, and adhesion is being conducted to determine the reproducibility of the results between different laboratories using different commercial testing equipment.

In electronic and optoelectronic multilayer structures, delamination driven by residual stress can lead to device failure. In collaboration with the NIST Optoelectronics Division, we characterize composition and structure in III–V optoelectronic materials and are developing Raman and photoluminescence techniques for measuring residual stress in films as thin as 30 nm. Three-dimensional finite-element simulations are being used to elucidate the influence of film properties and geometry on the residual stress tensor.

In many mechanical test methods, a force is applied to a specimen, and some displacement is measured. Traceable displacement measurement by interferometry is well established. Force measurement is more problematic because the Standard International (SI) unit for force is still based on an artifact kilogram mass. The Microforce Competence Program at NIST is developing a primary realization of force, traceable to electronic and length SI units, for force calibration in the range 1 mN to 1 nN. Transfer force cells are being developed that will allow force calibration, traceable to NIST, for commercial nanomechanical test equipment such as nanoindentation machines and atomic force microscopes.

### Contributors and Collaborators

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