

A New 'Wrinkle' in Nanometrology

Laminate buckling provides a new and robust metrology tool for nanoscale thin films.

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The quest to engineer materials on the nanoscale, for example ultrathin films, is challenged by the daunting task of measuring the physical and mechanical properties of these systems. Familiar techniques for measuring the mechanical properties of nanomaterials, such as nanoindentation, require expensive instrumentation, can be time-consuming, and exhibit poor reproducibility. In addition, these methods often are challenged by ultrathin films, soft materials such as polymers, and structured and/or filled materials such as nanocomposites. To overcome these obstacles, we have developed a rapid, robust measurement platform for probing the mechanical modulus (stiffness) of thin films and coatings materials, making it a useful characterization tool for combinatorial and high-throughput materials research.¹

Our technique leverages an elastic buckling instability that occurs upon compression of a film supported by a soft elastic substrate. The period of this buckling pattern (d) can be used to calculate the film modulus E_f using a well-established mechanical model for laminate systems:

$$\frac{E_f}{(1-\nu_f^2)} = \frac{3E_s}{(1-\nu_s^2)} \left(\frac{d}{2\pi h} \right)^3$$

This analysis scheme only requires knowledge of the substrate material's modulus E_s and Poisson's ratio ν_s , both of which are well known for common elastomers, and the thickness h and Poisson's ratio ν_f for the thin film. Polymers typically have Poisson's ratios of 0.25 to 0.35, so if you used 0.3 as a general approximation, the range of error is about 3% max.

We can determine the buckling wavelength by any one of several convenient techniques. If the buckles have periods of 1 μm up to 50 μm , the wavelength can be rapidly measured by laser light diffraction. Larger wavelengths can be captured using an optical microscope (see figure). Ripples with periods less than 1 μm can be imaged via atomic force microscopy (AFM). Light diffraction and optical microscopy are best for high-throughput workflow, since data acquisition and image analysis can be automated, providing single-point modulus measurements in less than 5 s; accordingly, this technique enables the rapid characterization of multivariate combinator-

ial film libraries. Acquiring images by AFM is inherently slower, but automating image analysis can still increase throughput.

Sample fabrication is a critical step in applying this buckling technique. For polymer materials, film specimens can be prepared on a separate substrate such as a silicon wafer, glass slide, or polished salt plate by spin coating, dip coating, solvent casting, doctor blading, or spray coating. To transfer the film onto the elastic substrate (we use cross-linked polydimethylsiloxane (PDMS)), we employ a water-immersion technique similar to floating films on water. Obviously, we cannot transfer water-soluble polymers this way; furthermore, PDMS swells in most organic solvents, so direct deposition of a polymer film onto PDMS is problematic unless a solvent-free technique is used. The technique works well for many materials, however.

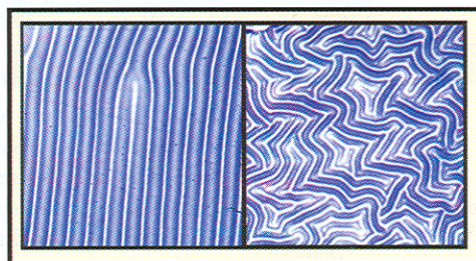
Although initially developed for polymer films, elastic buckling can help characterize a variety of materials systems, including metals, ceramics, and nano-structured composites. In collaboration with IBM (Armonk, NY), for example, we used this technique to measure nanoporous low- k films with pore dimensions of 5 to 50 nm. In addition, the method has successfully measured the elastic moduli of films as thin as 5 nm. In fact, this methodology works better for thinner films since thicker specimens are more rigid and require stronger adhesion to the substrate in order to suppress delamination of the film upon compression.

Our buckling modulus measurement platform is rapid, versatile, inexpensive to implement, and quantitative. These measurements work on a wide variety of materials systems and thin film geometries that are of interest to the nanotechnology community. We envision that this technique is positioned to become an important measurement tool for nanomaterials and nanosystems development. **oe**

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References

1. C. Stafford et al., *Nature Materials* 3 (2004).



Colorized optical micrographs of buckling patterns show the difference in patterns resulting from uniaxial (left) and isotropic (right) compressive strains.