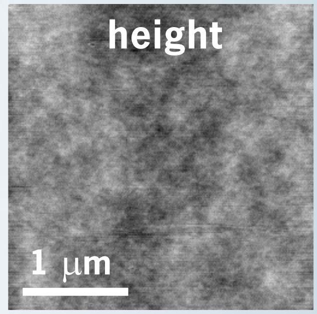
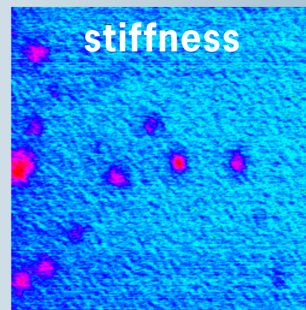
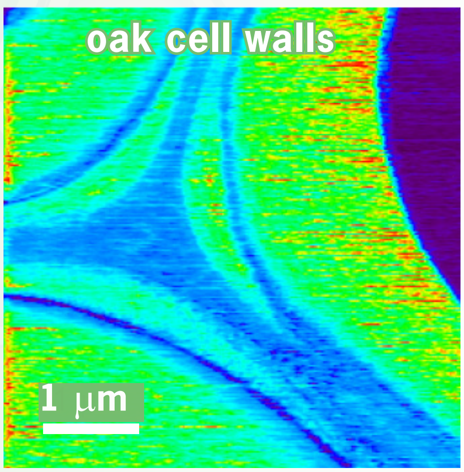
AFM-Based Nanomechanics

Objective

Local mechanical-property information is essential to evaluate emerging micro- and nanoscale materials, which many manufacturers would like to leverage for their unique properties. Existing methods, however, lack spatial resolution and do not visualize the distribution in properties. Our goal is to develop and apply new atomic force microscope (AFM) tools to rapidly map mechanical properties and features such as modulus, damping, adhesion and defects. Measuring localized variations in properties reveals material homogeneity and manufacturability and provides size-appropriate data critical for predictive modeling of device reliability and performance. Our methods can also be exploited for new types of AFM sensing to accelerate nanotechnology research and development.





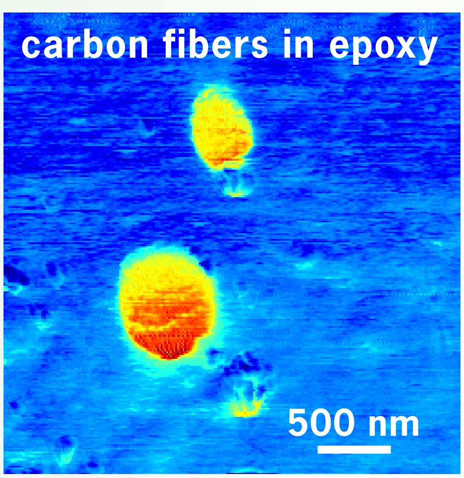


Impact and Customers

Nanocomposites are poised for explosive growth, particularly in the packaging, building and automotive industries. Enhanced composite performance requires optimal interfaces between the polymer matrix and the nanofiller. Nanomechanical mapping enables unprecedented levels of interface characterization and, ultimately, control.

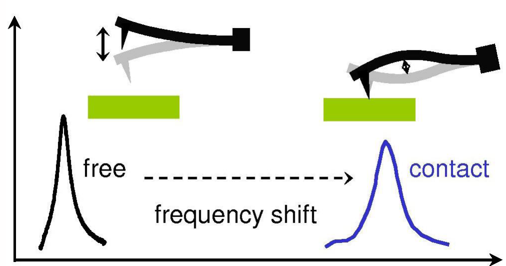
Development of new polymer products ranging from consumer goods to biorе-placement materials continues to accelerate. Many involve strongly viscoelastic, compliant materials with nano- and microscale heterogeneities. Evaluating the mechanical robustness of these materials is critical for qualifying future product reliability and performance.

New contact-based processes could revolutionize nanomanufacturing, but they cannot yet be sufficiently controlled to produce high-volume, low-cost products. Sensitive, real-time measurements of nanoscale contact interactions lead to enhanced nanofabrication process control and, hence, dramatic improvements in repeatability, yield and speed.



Approach

Originally developed for topography information, the AFM offers many advantages for materials characterization. Most notably, the small radius of the AFM tip (~5 nm to 50 nm) provides true nanoscale spatial resolution. Several AFM methods have been developed to assess mechanical properties, but typically only qualitative images of relative contrast can be obtained. In comparison, contact resonance force microscopy (CR-FM) enables quantitative mapping of mechanical properties. CR-FM involves measuring the frequency of the vibrating AFM cantilever while its tip is in contact with a sample. From these "contact resonance" frequencies, information is obtained about the interaction forces between the tip and the sample (e.g., contact stiffness). Models for the tip-sample contact mechanics are used to relate the contact stiffness to mechanical properties such as elastic modulus. Additional measurements of the contact resonance peak width (quality factor) allows the tip-sample damping interaction, and ultimately the sample's viscoelastic properties, to be determined. In systems with known mechanical properties, contact resonance signals are a rapid, sensitive probe of sub-nanometer changes in the tip-sample contact.



Accomplishments

In recent activities, we have continued to expand the measurement capabilities of CR-FM. With academic and industrial partners, we developed methods to determine viscoelastic properties from the frequency and quality factor of the contact resonance peak. We demonstrated this new approach on a blend containing polystyrene (PS) domains in a polypropylene (PP) matrix. Figure 1 shows viscoelastic CR-FM maps of the storage modulus (E') and loss modulus (E'') normalized to the mean PP values. There is relatively little difference in the stiffness (storage modulus) of the two polymers, but the damping (loss modulus) shows greater contrast. The maps in Figure 1 agree well with results obtained from dynamic mechanical analysis techniques on bulk samples. These results are promising for characterizing a wide range of industrially important, compliant materials.

In further work, we applied viscoelastic CR-FM methods to a biomaterial region containing stiff bone connected to compliant cartilage via mineralized cartilage. Mechanical-property information about this osteochondral interface is essential to reveal its functionality, understand its disease states, and develop reliable replacement materials. The CR-FM maps in Figure 2 of storage modulus and viscoelastic loss tangent reveal the spatial distribution of mechanical properties within the interface. With better spatial resolution than nanoindentation, CR-FM measurements indicated an interface width of a few micrometers and a gradual increase in loss tangent from the mineralized cartilage to the polymer-embedded cartilage. Our results not only provide insight into this specific interface region, but they also demonstrate the utility of CR-FM for a variety of systems containing interfaces between dissimilar materials.

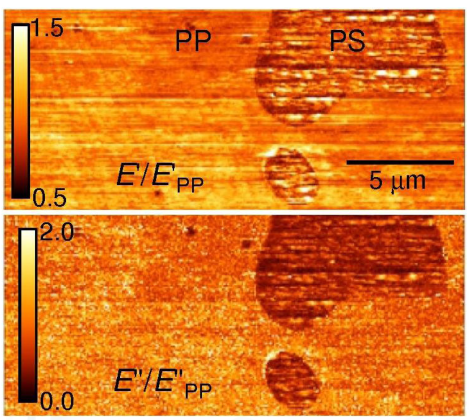
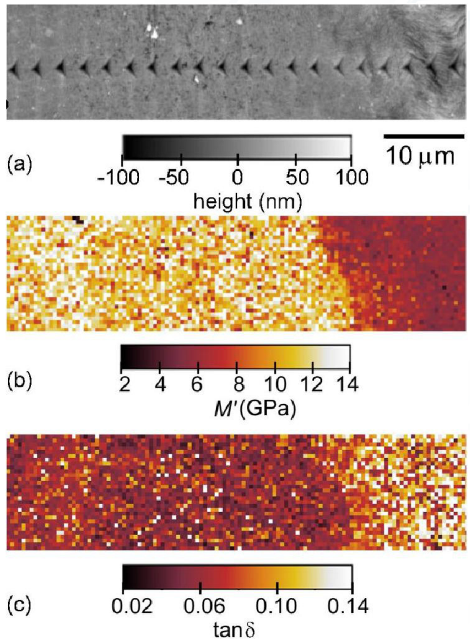


Figure 1. Maps of (top) relative storage modulus E'/E'_{PP} and (bottom) relative loss modulus E''/E''_{PP} for a PS/PP polymer blend obtained with viscoelastic CR-FM methods.

Figure 2. Maps of (a) topography, (b) storage modulus M′, and (c) loss tangent (tan δ) of bone-cartilage interface region obtained with CR-FM.



We have also begun to exploit contact resonance concepts for other nanotechnology research needs. For example, we have demonstrated how nanoscale wear of the AFM tip can be monitored *in situ* and in real time with CR-FM. As indicated in Figure 3, our approach provides continuous feedback as the tip is scanned across a smooth, uniform surface. In contrast, conventional approaches require intermittently interrupting the experiment to perform time-consuming imaging in the scanning electron microscope (SEM). This simple example represents a much broader class of nanomanufacturing operations that involve contacting, sliding interfaces. Use of CR-FM and related AFM methods to better understand these contact interactions will ultimately lead to nanofabrication processes with higher yield, better quality and improved commercial viability.

This work was publicized in over 20 invited and contributed presentations at national and international conferences and workshops in the last 18 months. We have written ten journal articles and two book chapters on our results in the same time period. In addition to reporting our technical results, we are engaged in efforts to transition CR-FM methods to a wider community. Technology transfer efforts include development of two software platforms for data analysis and substantial modification of our SPRITE module for improved data acquisition. We also taught lecture and laboratory modules in a three-day class on AFM nanomechanical measurements.

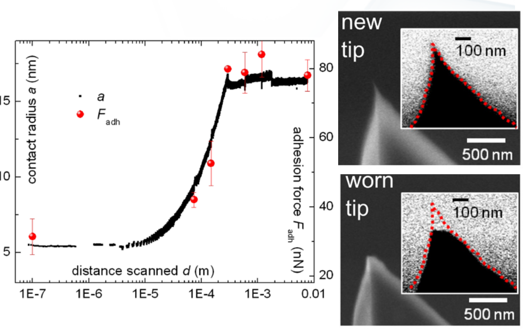


Figure 3. In-situ CR-FM measurements of contact radius (black line) continuously monitor the wear of the AFM tip during scanning and agree with intermittent AFM adhesion measurements (red dots) and ex-situ SEM measurements of tip shape (images).

Learn More

Donna Hurley, Jason Killgore, Sara Campbell, Tony Kos (PML)

Donna Hurley
303-497-3081
donna.hurley@nist.gov

http://www.nist.gov/mml/acmd

Publications

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JP Killgore and DC Hurley, Pulsed contact resonance for AFM nanomechanical measurements, Appl. Phys. Lett. **100** (5), 053104 (2012).

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