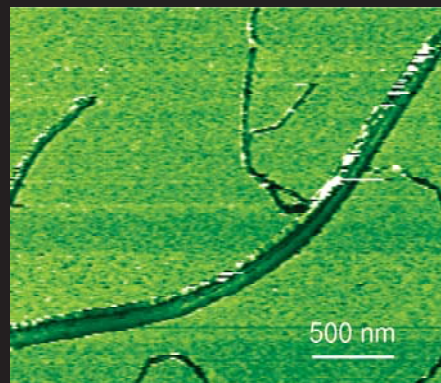


# AFM-Based Nanomechanics

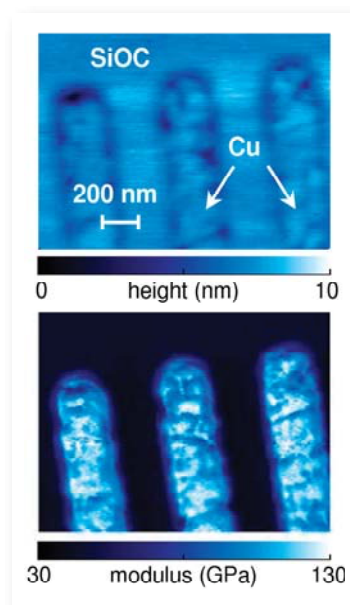
## Objective

Our goal is to provide tools for nanotechnology research and development that rapidly and nondestructively map the nanoscale mechanical properties of new materials and devices. Measuring localized variations in properties not only yields valuable information on material homogeneity and manufacturability, but also enables early identification of subsurface defects. Our methods also provide size-appropriate data critical for the predictive modeling of device reliability and performance.



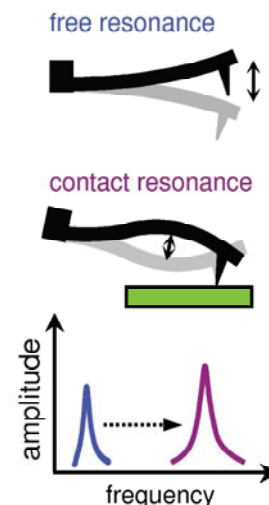
## Impact and Customers

- Nanoelectronics revenues approached \$11 billion in 2007 and should exceed \$80 billion by 2011 as manufacturers take advantage of the thermal management, miniaturization, and speed enabled by ultra-thin films and nanostructures. Determining the mechanical robustness of these materials is critical for qualifying future product reliability.
- Polymer nanocomposites are poised for explosive growth in the next few years, especially in the packaging, building, and automotive industries. Enhanced composite performance depends on creating ideal interfaces between the matrix and the nanofiller. Nanomechanical mapping enables unprecedented levels of interface characterization and, ultimately, control.
- Many nanoparticles are predicted to possess extreme mechanical properties, for instance very high strength in single-walled carbon nanotubes. However, characterizing this behavior remains difficult. Measurements at the single particle level will validate fundamental materials models.



## Approach

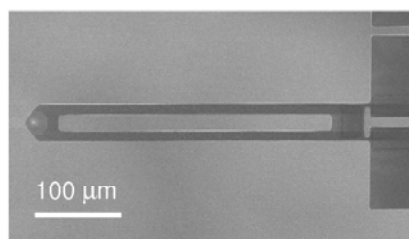
The atomic force microscope (AFM) offers many advantages for nanoscale measurements. Most notably, the small radius of the AFM tip (~5 nm to 50 nm) enables true nanoscale spatial resolution. Several AFM methods have been developed to assess mechanical properties, but most can only produce qualitative images. In contrast, contact-resonance force microscopy (CR-FM) enables quantitative mechanical-property mapping. CR-FM involves vibrating the AFM cantilever while its tip is in contact with a sample. In this way, the resonant modes of the cantilever—the “contact resonances”—are excited. From measurements of the 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 then used to relate the contact stiffness to mechanical properties such as elastic modulus.



## Accomplishments

Previous project activities focused on establishing the basic measurement methodologies so that CR-FM could be used as a quantitative tool. We also developed new techniques to enable quantitative imaging or mapping of mechanical properties. More recently, project activities have involved extending the basic concepts to achieve new measurement capabilities, and the use of CR-FM in new applications.

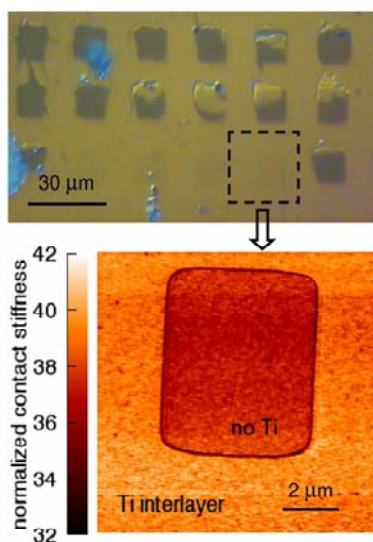
For example, we developed new methods to calibrate the spring constant of AFM cantilevers. Our approach involved a piezosensor transfer standard that was calibrated to absolute SI forces. If used appropriately, the piezosensor method yielded values accurate to ~5-10 %. This method is not affected by the cantilever geometry, eliminates calibration of the photodiode detector, and mimics the loading conditions of use. We also modified CR-FM methods developed for stiff materials in order to measure compliant polymers. We created an analysis approach to extract viscoelastic



Piezoresistive cantilever used as a force transfer standard.

properties from the amplitude and phase of CR-FM spectra. Values for the storage and loss moduli of a PMMA film were compared to those obtained from other techniques. The results show promise for a new methodology to measure compliant materials with nanoscale resolution.

In other experiments, we investigated the potential of CR-FM methods to evaluate mechanical properties besides modulus. A model sample was created with a gold blanket film over a patterned titanium interlayer on silicon. Scratch tests indicated that the film/substrate adhesion was much stronger in regions containing the



Optical micrograph (top) and CR-FM contact-stiffness map (bottom) showing variations in film/substrate adhesion.

interlayer. CR-FM images of the sample indicated that the contact stiffness was consistently lower by ~5 % in the regions with poor adhesion (no interlayer). The results represent progress towards quantitative imaging of adhesion, a goal with important technological implications.

Most recently, we developed CR-FM methods to quantitatively measure shear elastic properties such as Poisson's ratio  $\nu$  or shear modulus  $G$ . By measuring the contact-resonance frequencies of both the flexural and torsional modes of the cantilever,  $G$  or  $\nu$  can be determined separately from Young's modulus  $E$ . Experiments on a glass specimen were performed to demonstrate the validity of the approach. This new method means that further information about nanoscale mechanical properties can be obtained.

We reported this work through invited lectures at the Materials Science & Technology and the Materials Research Society national meetings, and at the American Physical Society March meeting. We have recently written a book chapter containing a "user's guide" to performing and optimizing CR-FM experiments that will be published in FY09. In FY08, we will pursue new uses for CR-FM including nanocomposites and film delamination. We are also working to extend CR-FM to enable detection of nanoscale subsurface defects.

## Learn More

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## Publications

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Langlois ED, Shaw GA, Kramar JA, Pratt JR, Hurley DC, *Spring constant calibration of AFM cantilevers with a piezosensor transfer standard*, Rev Sci Instr 78:093705 (2007)

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Hurley DC, Kopycinska-Müller M, Langlois ED, Kos AB, Barbosa N, *Mapping substrate/film adhesion with contact-resonance-frequency atomic force microscopy*, Appl Phys Lett 89:021911 (2006)