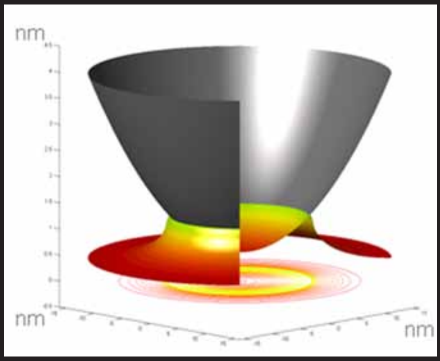
Scanning Probe Microscopy Measurements and Standards

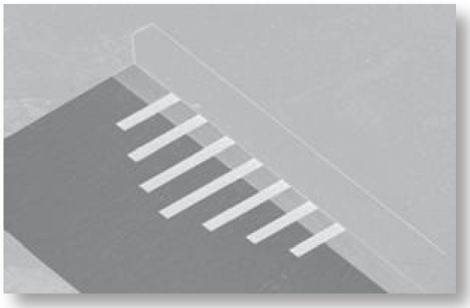
Objective

Our goal is to develop standard reference materials and quantitative, reproducible, measurement methods and protocols for scanning probe microscopes, to enable accurate dimensional, force, and material property measurements at the nanoscale. For example, our approach will allow force measurements in atomic force microscopes to be both precise and accurate.



Impact and Customers

- A broad spectrum of industries, government agencies, and academic institutions use scanning probe instruments to develop, characterize, and manufacture products from ceramics, metals, polymers, and semiconductors.
- Atomic Force Microscopes (AFMs) are the most common scanning probe instrument, with an estimated 10,000 AFMs in use in virtually every materials research and development laboratory worldwide.



- Calibrating delicate measurement tools such as AFMs is difficult: at small length scales, the forces that affect probe-material interactions, and their relative magnitudes, are often unknown, but are certainly dominated by surface effects.
- Currently, the lack of SI-traceable stiffness calibration standards hampers progress towards making AFM force measurements quantitative; such measurements can be precise, but there is an incomplete understanding of accuracy.

Approach

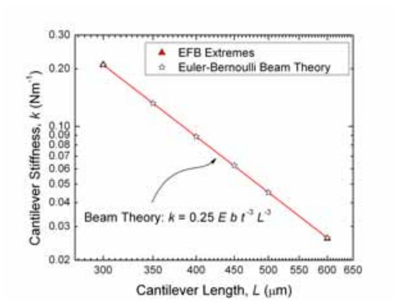
A NIST Standard Reference Material (SRM 3461) will be produced that will enable accurate calibration of the flexural stiffness of AFM test cantilevers. An array of extremely precise rectangular cantilevers will be microfabricated from silicon-on-insulator wafers. The uniformity of the cantilever population on each wafer will be verified with resonance frequency measurements using Laser Doppler Velocimetry and Euler-Bernoulli modeling. Calibrations traceable to the International System of Units (SI) will be performed on a statistical subset of the population using the electrostatic force balance developed by the Manufacturing Engineering Laboratory at NIST.

Further, AFM methods will be developed to enable mechanical property measurements at the nanoscale. Contact resonance methods will be used to determine elastic properties. AFM experiments using conducting probes have demonstrated the ability to measure thin interfacial oxide and organic films.

NANOMATERIALS

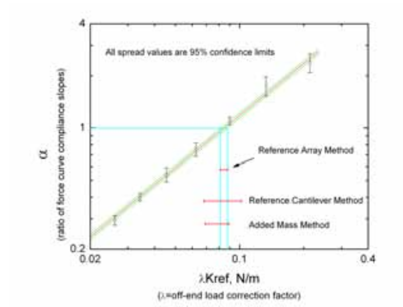
Accomplishments

A series of extremely uniform prototype reference cantilever arrays were created that can be used to calibrate the spring constants of atomic force microscopy cantilevers and other micromechanical structures. Nominal spring constants were estimated to be in the range of 0.02 Nm^{-1} to 0.2 Nm^{-1}. Resonance frequency measurements were used to assess the uniformity of devices from different portions of a silicon-on-insulator wafer, and from different processing batches. Variations of less than 1% (relative standard deviation) in resonance frequency attested to the high degree of uniformity achieved. Independent calibration of cantilevers in an array using an electrostatic force balance indicated that the actual spring constants ranged from 0.0260 ± 0.0005 Nm^{-1} (±1.9%) to 0.2099 ± 0.0009 Nm^{-1} (±0.43%). The results confirmed the feasibility of creating uniform reference cantilevers and calibrating them using an SI-traceable technique, making these devices excellent candidates for small force calibration standards for AFM. An SRM production batch is currently in process.



Agreement between stiffness measurements and models

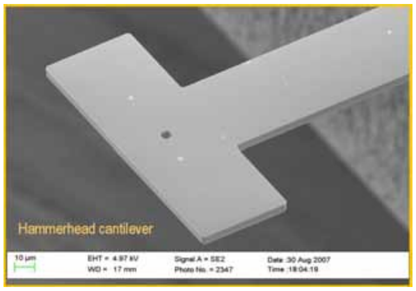
A method for calibrating the stiffness of AFM cantilevers was developed using the prototype reference cantilever array. A series of force-displacement curves was obtained using a commercial AFM test cantilever on the reference cantilever array, and the data were analyzed using an Euler-Bernoulli model to extract the test cantilever spring constant from linear regression fitting. The method offers improvements in precision over the reference cantilever method (factor of five) and the added mass calibration method (factor of three) that are currently used for AFM calibration.



Comparison of calibration methods

A new "Hammerhead" AFM cantilever has been developed that is capable of providing precise lateral force (e.g. friction) measurements on surfaces using a commercial instrument. The "wings" projecting from the sides of the cantilever are used along with a specially designed calibration chip that allows for precise determination of the optical torsional sensitivity of the photodetector, which is usually very difficult to measure. A production batch of reference material "Hammerhead" cantilevers is being developed and fabricated to make these devices available to the research community.

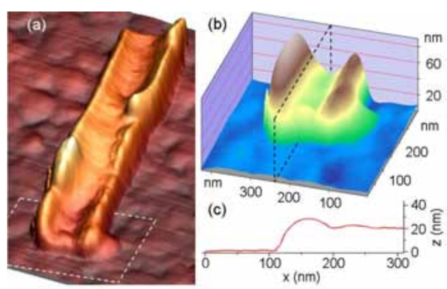
A new, higher-precision contact-resonance AFM (CR-AFM) method was developed to



Prototype "Hammerhead" cantilever for precise lateral force measurment in an AFM

quantitatively determine material indentation moduli by measuring local mechanical responses. A dual reference method has been shown to be capable of extracting the modulus of a material within 3% of the calculated expected value, without any assumptions of the probe's mechanical properties.

Using this CR-AFM technique, select nanostructures have been studied to elucidate their nanomechanical properties. The most recent work included analysis of ZnO nanowires as small as 26 nm in diameter, and AlN nanotubes 200 nm in diameter but with wall thicknesses of only 19 nm.



A split AlN nanotube used for morphology and contact resonance AFM analysis

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Publications

Gates RS and Pratt JR, *Prototype Cantilevers for SI-Traceable Nanonewton Force Calibration* Meas. Sci. Technol., 17: 2852 (2006)

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Kim DI, Namboodiri P, DelRio FW and Cook RF *Mechanical and Electrical Coupling at Metal-Insulator-Metal Nanoscale Contacts* Applied Physics Letters, 93: 203102 (2008)

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