Prototype Cantilevers for AFM Calibration and Nanomechanical Property Measurement

CNF Project # 1273-04

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Primary Research Funding: National Institute of Standards and Technology

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Abstract:

Atomic force microscopy (AFM) is a widely used technique for imaging surfaces and measuring properties at the micro and nano-scales; however, the accuracy and precision of these measurements is hampered by the lack of suitable traceable standards and precision measurement methods. The purpose of this project is to explore potential cantilever designs as calibration reference artifacts and probes for making precise nanomechanical property measurements.

Summary of Research:

This project investigates microfabrication using silicon-on-insulator (SOI) materials to create two types of cantilever devices. The first is a reference cantilever array that could be used to accurately calibrate the spring constants of AFM test cantilevers in the field. The second device is a cantilever that is modified with appendages at the free end to facilitate the measurement of the torsional optical sensitivity of the cantilever and make it useful for friction measurements. We call this latter device the “Hammerhead” cantilever.

Reference Cantilever Calibration Method:

Although there are several methods for calibrating AFM cantilevers, they are usually limited in scope to specific cantilever types or spring constants. The absolute accuracy of the methods is unknown since none of them are currently traceable to the Système International d’Unité (SI). One of the most widely applicable methods (reference cantilever method [1, 2]) relies on pushing the unknown cantilever against a cantilever of known stiffness and measuring the deflection. If the stiffness of the unknown cantilever is reasonably close (within a factor of 10) to the stiffness of the reference cantilever, the spring constant of the unknown cantilever can be calculated. Commercial reference cantilevers are available with nominal spring constant values but their accuracy is unknown since they have not been traced to the SI.

Standard Reference Cantilever Prototype:

The objective of this project is to investigate the feasibility of creating very accurate reference cantilevers that could be used to calibrate the spring constants of AFM cantilevers. This requires very uniform cantilevers that could be calibrated using an SI-traceable technique using statistical sampling. The key to cantilever uniformity lies in careful dimensional control during microfabrication. Since the spring constant \( k \) of an ideal, uniform, rectangular cantilever can be described by an Euler-Bernoulli model [3] (eq. 1) that depends on elastic modulus \( E \) and width \( b \) to the first power but the cube of the thickness \( t \) and length \( L \), it is especially important to control these last two characteristics.

\[
 k = \frac{Eb^2}{4L^3} \\
 f_{\text{vac}} = 0.1615 \frac{t}{L^2} \sqrt{\frac{E}{\rho}}
\]

Equation 1  
Equation 2

Initial prototypes were successfully microfabricated with SOI wafers using anisotropic etching and deep reactive ion etching in combination with patterning by e-beam lithography. An array of these cantilevers is shown in Figure 1 and consists of seven cantilevers 50 µm wide, 1.4 µm thick, and varying in length from 350 µm to 600 µm.

Since the cantilever resonant frequency, \( f_{\text{vac}} \), (eq. 2), depends on the same key parameters of thickness and
length as the spring constant (just to different powers), resonant frequency was used as a measure of uniformity of the cantilevers from different parts of a wafer. The standard deviation of the mean resonant frequency for 22 cantilever arrays was less than 1.0%, suggesting excellent uniformity control. The spring constants of cantilevers made by this process were also measured using a special apparatus designed and fabricated at NIST to provide SI traceable force calibration at the nN scale [4] with a precision of 2 % for a stiffness as small as 26 pN/nm. The results confirm the feasibility of microfabricating SI-traceable reference cantilevers that could be made available to the AFM community; however, the low yield of the process forced a change in the microfabrication process. A new set of prototypes are currently being fabricated using reactive ion etching and deep reactive ion etching in combination with stepper-based optical lithography.

Hammerhead Cantilever Prototypes:
The second prototype cantilevers, intended for enabling precise nanomechanical property measurements are the Hammerhead cantilevers. These devices consist of cantilevers patterned using contact lithography and etched out of SOI (Figure 2) using deep reactive ion etching. The two side appendages at the end of the cantilever facilitate optical lever calibration of the device so that precise lateral force measurements (e.g. friction) can be made. Figure 3 shows the other end of the AFM chip that contains a cutout and special fiducials that allow accurate alignment of the cantilevers to the cutout during the critical optical sensitivity calibration.

The microfabricated Hammerhead prototypes are currently being evaluated for their ability to measure the torsional optical lever sensitivity precisely using refinements in the new calibration method. Torsional spring constants, measured using a calibrated instrumented indenter, have compared favorably with Euler-Bernoulli theoretical models based on dimensional measurements and material properties for these cantilevers. Experiments have also been conducted using Hammerhead cantilevers with spheres attached to the end for friction measurements.

References: