Precise atomic force microscope cantilever spring constant calibration using a reference cantilever array

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A method for calibrating the stiffness of atomic force microscope (AFM) cantilevers is demonstrated using an array of uniform microfabricated reference cantilevers. 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 implied Euler-Bernoulli model to extract the test cantilever spring constant from linear regression fitting. The method offers a factor of 5 improvement over the precision of the usual reference cantilever calibration method and, when combined with the Système International traceability potential of the cantilever array, can provide very accurate spring constant calibrations. © *2007 American Institute of Physics.*
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The accurate measurement of spring constants for atomic force microscope (AFM) cantilevers is important for a variety of AFM applications, and for more than a decade, a number of techniques have been proposed and refined to estimate normal cantilever stiffness.[1–10] The use of a reference cantilever[4–6] as a calibration artifact is especially attractive as it can be used for many different types of cantilevers including both triangular and rectangular cantilevers, coated cantilevers, and colloid probes: it does not require an accurate knowledge of the cantilever dimensions or material properties and the range of force application is only limited by the availability of suitable (similar spring constant) reference cantilevers. Two problems that limit the accuracy and precision of the reference cantilever calibration method are the lack of accurate (Système International or SI traceable) standard cantilevers and the limited repeatability of the usual single point reference calibration procedure,[6] currently estimated as $\pm 10\% - \pm 30\%$.[10] Recently, an experimental reference cantilever array that offers the potential to be an SI traceable force transfer artifact[11] through calibration by an electrostatic force balance (EFB) has been produced.[12] In the present article we describe a method for using such a cantilever array to calibrate the spring constant of an unknown AFM cantilever more precisely.

The basic principle of calibrating the normal spring constant of an unknown test cantilever using a calibrated reference cantilever is depicted in Fig. 1. The calibration is performed by measuring the deflection of the test cantilever as it is pressed against a known reference force (the reference cantilever deflected a known amount at a specific location on the reference cantilever beam). For optical lever type AFM's, this is practically accomplished by making two force-displacement measurements: one on a rigid surface (well approximated by a stiff, smooth, bulk Si surface) and the second on a known location on the reference cantilever. Essentially, the first measurement establishes the relationship between photodiode (detector) voltage and piezoelectric displacement actuator motion in the z direction through the test cantilever deflection. The second measurement relates this motion to the deflection of the test cantilever on the reference cantilever. Typical approach force curves obtained on both a stiff surface and a reference cantilever are shown in Fig. 2 and consist of the approach to surface (a), snap-on (b), and compliance (c) regions in which the tip of the cantilever is moving with the surface it is contacting. The test cantilever displacements on the stiff surface and reference cantilever can be obtained from the slopes (S, V/nm) of the compliance regions of the force curves for the respective surfaces (a linear regression fit for data within the compliance region is used to determine these values). Correcting for the off-end loading of the reference cantilever,[9] we can use the compliance slopes to obtain the test cantilever spring constant through Hooke's law and the Euler-Bernoulli beam equation[13] as

$$k_{\text{test}} = k_{\text{ref}}\left(\frac{L}{L - d_{\text{load}}}\right)^3\left(\frac{S_{\text{stiff}}}{S_{\text{ref}}} - 1\right)\cos^2\theta, \qquad (1)$$

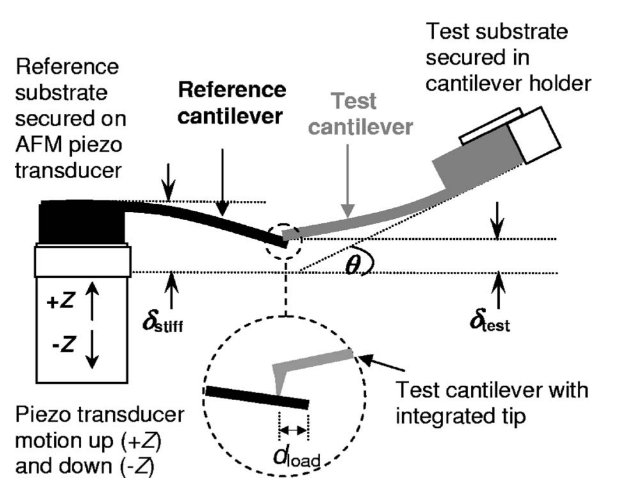

FIG. 1. Schematic diagram of the AFM configuration used for measuring the spring constant of an unknown test cantilever using a calibrated reference cantilever.

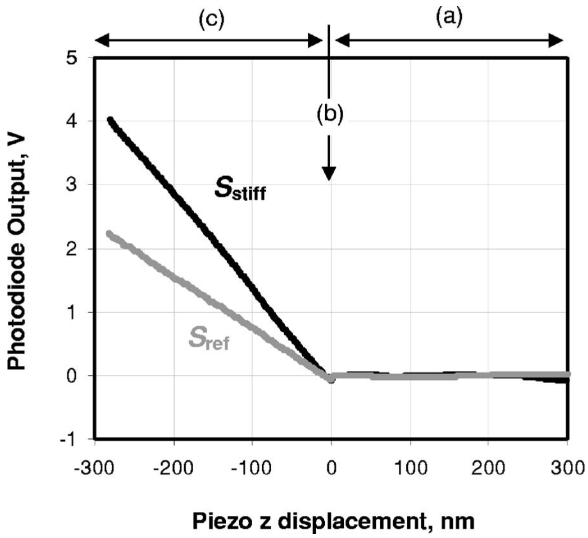

FIG. 2. Force-displacement curve examples (approach only) for a test cantilever contacting a stiff surface (bulk Si) and a reference cantilever

where k_{ref} and L represent the spring constant and length of the reference cantilever and d_{load} is the off-end loading distance from the actual point of contact on the reference cantilever to its free end. The $\cos^2\theta$ term is a geometric correction for the tilt angle of the test cantilever. In the case of the 11° tilt for our AFM, there is less than a 4% correction ($\cos^2\theta=0.9636$). Thus we can obtain an estimate of the test cantilever spring constant through the ratio of compliance slopes without explicit measurement of the photodiode or actuator responses.

All experiments were carried out on a Digital Instruments[14] Nanoscope IIIa AFM. The test cantilever used was a Veeco DNP type triangular cantilever ("D" = long, thin legged), which was calibrated independently via a five bead Cleveland added mass calibration with off-load correction,[1,9] giving a normal spring constant of 0.079±0.005 N/m.

The experimental reference cantilever array (Fig. 3) was microfabricated from a Si (100) silicon-on-insulator (SOI) wafer in such a way as to provide a high degree of dimensional control, pattern registration, and uniformity.[11] Each array consisted of seven cantilevers of identical thicknesses (1.4 μm) and widths (50 μm) and varying from 300 to 600 μm in length. They were considered as uniform rectangular Euler-Bernoulli beams. Nominal spring constants, estimated from dimensional and resonance frequency measurements and material properties, varied from 0.203 to 0.207 N/m, depending on the length of the cantilever selected. The values agreed well with the $k \propto L^{-3}$ behavior implicit in Euler-Bernoulli beam theory for uniform rectangular cantilevers, as well as with the independent calibration of an identical cantilever array by EFB.[11]

Using the commercial test cantilever, a series of eight (repeat) pairs of force-displacement curve measurements (Si substrate and reference cantilever) were conducted on each reference cantilever with the results summarized in Table I. The estimated spring constant for the test cantilever ranged from 0.077 to 0.091 (average of 0.085) with a relative standard deviation (all data) of approximately ±10%. The larger uncertainty for the shorter cantilevers is attributed to relative

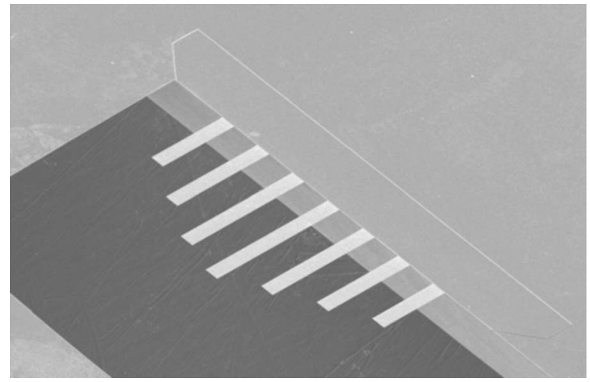

FIG. 3. Scanning electron micrograph of the experimental reference cantilever array. The cantilevers are nominally 1.4 μm thick and 50 μm wide and vary in length from 300 to 600 μm.

placement uncertainty (d_{load}) which contributes geometrically (to the third power) to the error.

Data taken using a range of reference spring constants provide an opportunity to estimate the spring constant of the test cantilever in a more precise way. Equation (1) can be rewritten in terms of the off-end-load correction term λ and a compliance slope ratio parameter α as

$$\alpha = \frac{\lambda k_{ref}}{k_{test}}, \tag{2}$$

where

$$\alpha = \left[\left(\frac{S_{stiff}}{S_{ref}} - 1\right)\cos^2\theta\right]^{-1} \text{ and } \lambda = \left(\frac{L}{L-d_{load}}\right)^3.$$

If we then plot the experimental dependent variable α versus the independent variable λk_{ref} we should get a straight line with slope $1/k_{test}$. In some ways this is analogous to the Cleveland method[1] in which different masses are added to the cantilever and the spring constant is extracted from the slope of the plotted data. In our case, we use different stiffness springs. The same data used to generate Table I are plotted in Fig. 4 as α vs λk_{ref}. The slope of the linear regression best fit provides a k_{test} of 0.085 N/m. Statistical analysis

TABLE I. Test cantilever calibration series on individual reference cantilevers

Array cantilever property		Single point reference calibration		
L (μm)	k_{ref} (N/m)	k_{test} (N/m)	±1 sd (N/m)	rsd (%)
300	0.207	0.091	0.012	13
350	0.130	0.077	0.011	14
400	0.088	0.081	0.005	6
450	0.061	0.084	0.008	9
500	0.045	0.085	0.008	9
550	0.034	0.086	0.004	5
600	0.026	0.089	0.006	6
		Average 0.085		

sd = standard deviation
rsd = relative standard deviation

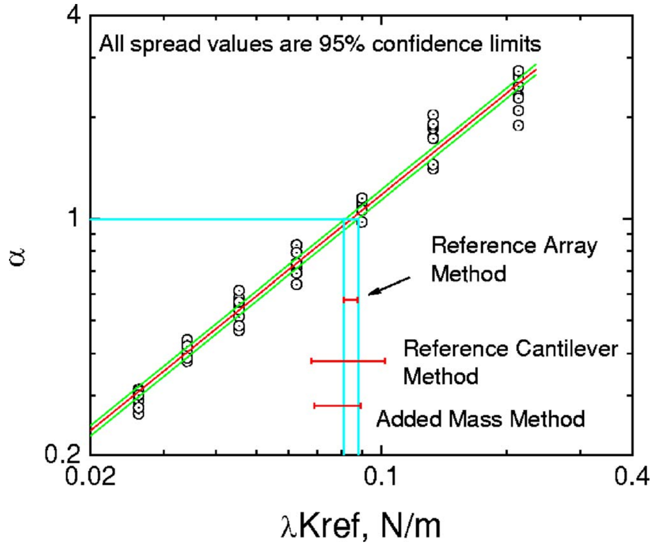

FIG. 4. (Color online) Reference cantilever array plot for a DNP-D test cantilever. Repeatabilities (±2 standard deviations) are compared to a single reference cantilever method and an added mass method.

of the linear regression provides an uncertainty (standard error) of the slope of only ±2%. This uncertainty in the determination of k_{test} is a substantial improvement over both the single point reference cantilever method (±10%) and the Cleveland method (±6%).

A second way of graphically extracting the stiffness and precision of the test cantilever is to use the same data in Fig. 4 and superimpose 95% confidence limits. The value of k_{test} (0.085 N/m) can be read off the graph at the point where $\alpha=1$. When this is done for the upper and lower bounds on the 95% confidence limits, we get a graphical representation of the precision of the data. Precisions of the single point reference cantilever and added mass methods are superimposed on Fig. 4 as a visual comparison. It should be noted that the key to the array cantilever method is the implicit adherence of the data to the Euler-Bernoulli model allowed by using the extremely uniform cantilevers in the array.

The data examined in Fig. 4 represents an optimal case since the spring constant lies midway along the array specifications; however, we have also conducted exploratory experiments using test cantilevers near the extremes of the array spring constants. Using a stiff test cantilever ($k=0.65$ N/m) also provided very good precision (2%). Using a very compliant test cantilever ($k=0.014$ N/m) we found significantly poorer precision (8%) due to variations in the force curve behavior. We suspect that this was due to complications from the buckling and twisting of the test cantilever tip. In both extremes the array method precision was still better than that of the regular reference cantilever method by a factor of 4.

Using an array of reference cantilevers with different spring constants we have demonstrated that it is possible to make measurements on the array and analyze the data in such a way as to obtain a spring constant for an unknown cantilever with greater precision than possible with just repeated measurements on a single cantilever or even averaged measurements on several cantilevers. When this approach is combined with independent validation of the reference cantilevers with an SI traceable method, this technique offers an accurate and precise method for calibrating AFM cantilevers in the field.

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[14] Certain commercial equipment, instruments, or materials are identified in this article to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.