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CALIBRATION OF MICROFABRICATED CANTILEVERS FOR SI-TRACEABLE SMALL FORCE MEASUREMENT

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ABSTRACT: A procedure is described by which the spring constant of a microfabricated cantilever beam can be calibrated for the measurement of small forces in an atomic force microscope (AFM) or other device. The procedure utilizes dynamic force instrumented indentation to determine the mechanical properties of the beam by applying a well-characterized oscillating force and measuring resulting displacement of the system. An uncertainty analysis is carried out, and by intercomparison with the U.S. National Institute of Standards and Technology (NIST) Electrostatic Force Balance (EFB). The spring constants determined using the indentation method agree within 2 % of the values determined using the EFB for spring constants as low as 2 N/m.

KEY WORDS: Atomic force microscopy, force, nanotechnology

1. INTRODUCTION

The atomic force microscope (AFM) is an unusual instrument in that it allows characterization of both topographical features and mechanical properties of a surface. Central to the accurate interrogation of the latter is the probe used in the instrument. Typically, this probe is a microfabricated cantilever beam, and the force it applies can be calculated by measuring the deflection of the beam, and assuming it behaves as a linear spring as in Hooke's law:

$$F = kX \tag{1}$$

where F is applied force, X is the deflection of the cantilever beam at the point of application of the force, and k is the spring constant of the system. This approach requires an accurate value for k if force metrology is desired.

Various methods have been developed to determine k [1], each with their own set of strengths and weaknesses. None of these methods currently provide a direct link to the SI. In order to facilitate a link to the SI, F and X can be measured directly in a traceable fashion to yield a traceable value for k. However, since the cantilevers are typically quite small (on the scale of 10 μm to 100 μm), this presents significant experimental challenges.

In this study, dynamic force instrumented indentation was used to determine the spring constants of a microfabricated cantilever. An intercomparison was provided by using the NIST electrostatic force balance (EFB) to perform quasi-static SI-traceable measurements of applied force and resulting displacement of the cantilever.

2. METHODS

The cantilever under test was designed specifically for the purpose of spring constant calibration [4]. It was fabricated from polycrystalline silicon to be approximately 1600 μm long, 150 μm wide and 3 μm thick. There were lithographically-defined fiducial marks along the cantilever's length which could be used to locate a specific point for testing. Since the spring constant of a cantilever scales with length^{-3}, this allowed for a wide range of spring constants to be tested.

Instrumented indentation measurements were performed with a commercial instrument, and are illustrated in Figure 1. The force measurement of this instrument had been calibrated previously using an auxiliary load cell [2]. Displacement was interferometrically calibrated by the vendor. The use of a proximal optical microscope and an encoded translation stage allowed placement of the cube corner indenter tip within 2 μm of the desired test location. The spring constant of the indentation transducer was first determined by applying a sinusoidally varying force to the indenter and measuring the resulting displacement with a commercial lock-in amplifier. Assuming the system behaves as a single degree of freedom harmonic oscillator, the spring constant of the indentation transducer, k_i, can be calculated as

$$k_i = \frac{F}{X}\cos\phi - m\omega^2 \tag{2}$$

where φ is the phase angle between applied force and resulting displacement, m is the system mass, and ω is the test frequency. The indenter is then lowered into contact with the cantilever under test, and the same test is performed to yield the measured spring constant of the combined system, k_m. The spring constant of the cantilever under test, k_c, is then determined using

$$k_c = k_m - k_i \qquad (3)$$

Tests were performed at a frequency of 10 Hz, at a force amplitude of 7.5 μN, and a preload of 10 μN.

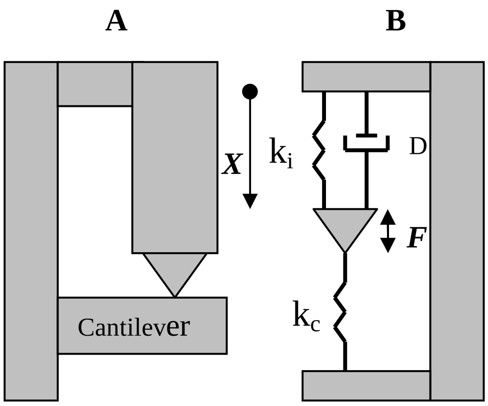

Figure 1. Illustration of the experiment used to determine k. The experimental configuration is shown in A. A sinusoidally varying force calibrated with deadweights is applied to the cantilever, and displacement is monitored with a lock-in amplifier. The mechanical block diagram of the system is shown in B, and the variable D is the viscous damping of the system.

The operation of the EFB has been previously described [3]. In order to calibrate AFM cantilever spring constants, a spheroconical indenter tip was attached to the moving arm of the balance. SI-traceable electrostatic forces were then applied to the indenter tip as its displacement was monitored interferometrically. This provides a direct measurement of an SI-traceable spring constant.

3. RESULTS

The spring constants and their associated uncertainties for the cantilever under test as determined by the dynamic indentation method and the EFB are presented in Table 1 for the 3 points tested. Points 1, 2, and 3 were located 172 μm, 412 μm, and 712 μm from the cantilever base, respectively.

Table 1. Cantilever Spring Constants.

Test Point	k_c indentation (N/m)	k_c EFB (N/m)
1	18.90 ± 0.32	18.883 ± 0.015
2	2.03 ± 0.24	2.0656 ± 0.003
3	0.40 ± 0.18	0.461 ± 0.0019

4. DISCUSSION

A standard deviation of 0.044 N/m was measured during the determination of k_m if the system was closed and allowed to equilibrate overnight. In order to determine k_c using Equation 3, k_i must also be measured, and its standard deviation was found to be 0.062 N/m. These type A uncertainties were determined by calculating the standard deviation of each set of 100 data points used to measure k_m and k_i, and then determining the mean value of this standard deviation over the course of the three trials conducted. Summing these uncertainties in quadrature [5] results in a combined standard type A uncertainty of 0.076 N/m.

Equation 2 requires accurate force and displacement measurements. Previous work has demonstrated the use of an auxiliary capacitive load cell for indenter force calibration based on deadweight forces [2]. This allows measurement of force with an uncertainty of 1 %. Displacement sensitivity of the indentation transducer was calibrated by the vendor using an interferometer. Since the value of k_i determined from the dynamic test results gives agreement with the vendor's specification within 1%, the maximum displacement uncertainty was estimated to be 1%. Since the uncertainties in force and displacement are systematic, they are determined by non-statistical methods (i.e. type B uncertainties). As above, these uncertainties must be counted twice when calculating combined standard uncertainty because they are present when measuring both k_m and k_i for use in Equation 3. Likewise, they are summed in quadrature with the other uncertainties present during the measurement in Table 2.

The applicability of equation 1 depends on the linear behavior of the elastic element. Since deviations from the linearity expressed in Equation 1 can arise if a cantilever is bent too far, care must be taken to keep deflection within a maximum of 5 % of cantilever length. During the calibration, oscillating and bias forces of 7.5 μN and 10 μN , respectively, were applied against the combined stiffness of the cantilever, and the internal spring of the indentation sensor which has stiffness of k_i = 153 N/m. The total stiffness of the system is $k_c + k_i$, which ranges from 154 N/m to 173 N/m. This results in DC displacements of approximately 60 nm, and AC displacements of approximately 40 nm peak-to-peak amplitude in the combined cantilever/transducer system. This is well within their linear regimes of the cantilevers tested in all cases.

The cosine and inertia terms in Equation 2 contribute much less to the uncertainty. The phase angle (φ) was measured to be less than 0.1° in all experiments. This results in a maximum standard uncertainty of 2 x 10^{-6} of the value of k, and is considered negligible for the purposes of this uncertainty analysis.

Equation 2 also includes an inertial term ($m\omega^2$). The approximate mass of the cantilever tested for this study is 1 µg. The mass of the indenter tip was measured to be approximately 18 mg, so the increase in system mass upon contacting the cantilever beam is negligible. The vendor's specification for uncertainty in frequency is 3 x 10^{-6} Hz. Since the same frequency is used to determine k_m and k_i, the inertia term ($m\omega^2$ in Equation 2) can be eliminated as a common mode term in Equation 3, with a negligibly small increase in uncertainty.

The location of the point of contact between the indenter and reference cantilever must also be specified precisely to minimize uncertainty. Assuming the reference device behaves as an ideal Euler-Bernoulli beam, the spring constant scales with the inverse cube of the distance from the cantilever base to the test point, so uncertainty due to indenter placement can be calculated using

$$u_p = 3\left(\frac{\Delta l}{l_t}\right)k \qquad (4)$$

where Δl is the uncertainty in indenter tip placement (approximately 2 µm), and l_t is the distance from the base of the cantilever to the test point. The values of l_t are 172 µm, 412 µm, and 712 µm for test points 1, 2, and 3, respectively.

The sources of uncertainty, their magnitudes, and the combined standard uncertainty [5] of the dynamic indentation results shown in Table 1 are summarized in Table 2.

Table 2. Sources of Uncertainty in Calibration.

Source of uncertainty	**Magnitude of uncertainty**
Statistical uncertainty	0.076 N/m
Displacement measurement	0.01 k
Force measurement	0.01 k
Position of indenter on cantilever under test	$u_p = 3\left(\frac{\Delta l}{l_t}\right)k$
Combined Standard Uncertainty	$[0.076^2 + 4(0.01\,k)^2 + u_p^2]^{1/2}$

In the EFB, a traceable electrostatic force is realized directly through measurements of SI electrical quantities with relative standard uncertainties on the order of 1 part in 10^5. Likewise, displacement is measured interferometrically with 5 nm uncertainty. The effect of the uncertainty in position of the contact between the indenter tip and cantilever is not included in the EFB results in Table 1.

Ultimately, these cantilevers will be used as reference springs to calibrate the spring constants of other cantilevers for quantitative force measurement in AFM. Although a transfer of these traceable reference spring constants to an AFM cantilever have been demonstrated [6], there are difficulties in maintaining a high level of precision and accuracy in the transfer. The major issues appear to stem from the mechanics of the contact between the AFM and reference cantilevers. More specifically, the AFM cantilevers are deviating from the ideal behavior described by equation 1 as a result of off-axis forces. These are forces that are not normal to the surface plane of the AFM cantilever, and can result from the friction of the sliding contact between the AFM and reference cantilevers [7], or from the AFM cantilever's angle of repose [8]. These effects must be clearly understood in order to minimize additional uncertainty arising from the transfer process.

5. CONCLUSION

Methodology for the SI-traceable determination of AFM cantilever spring constants has been established by two different means. The methods agree within experimental uncertainty, and provide a route for SI-traceable small force measurements using microfabricated cantilevers.

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