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Experimental determination of scanning probe microscope cantilever spring constants utilizing a nanoindentation apparatus

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A rapid, nondestructive, and accurate method for determining the normal spring constants of scanning probe microscopy cantilevers is presented. Spring constants are determined using a commercial combination atomic force microscope and nanoindentation apparatus configured with a W-indenter tip geometrically configured into either a scanning tunneling microscope pointed tip or chisel shape that may be placed onto the cantilever of interest with high accuracy. A load is applied to the cantilever tip and the corresponding displacement is measured. From the force–displacement curve, the spring constant is determined. For cantilevers with spring constants greater than 1 N/m, the derived spring constants are believed to be accurate to within $\pm 10\%$, with better accuracy for stiffer levers. This method has been used to measure the stiffness of cantilevers from several manufacturers. © 2000 American Institute of Physics. [S0034-6748(00)01010-8]

I. INTRODUCTION

A. Calibration techniques

Atomic force microscopy (AFM) has proven to be an effective tool for the quantitative measurement of nano-Newton (nN) scale forces in numerous systems.^{1–8} To extract force data from AFM measurements, the position-sensitive photodiode detector signal must be converted into a displacement of the cantilever tip. This displacement is multiplied by the spring constant to obtain a force. Cantilever spring constant uncertainty is recognized as a major source of systematic error in performing quantitative AFM force measurements.⁹ While published spring constant values of microfabricated cantilevers with integrated tips exist from all manufacturers, the spring constants on even the most accurate commercial cantilevers on the market have uncertainties of more than $\pm 50\%$.¹⁰

Several research groups have developed methods to determine AFM cantilever spring constants, although each exhibits a specific drawback, either in its application, inherent test complications, destructiveness, or lack of accuracy. Generally, methods are divided into three groups: dynamic methods based on determining the cantilever resonance; methods that measure deflection upon static loading of the lever; and methods that calculate the spring constant from the physical dimensions of the lever as measured by scanning electron microscopy (SEM).¹¹

Methods based on dynamic response utilize the scanning

force microscope's ability to determine the resonant frequency of a particular lever, either with an added mass or subjected to thermal excitation. Measurement of the change in cantilever resonance frequency upon the addition of an added mass has a claimed accuracy of 10%–15%, although this is highly dependent on the location of the mass.¹² This technique requires the attachment of spheres of known mass to the cantilever using a three-dimensional micromanipulator and is potentially destructive.¹³ Additionally, thermal fluctuations can be used to stimulate the principal vibration mode. While this method is reported to be reliable for soft levers ($k_N < 0.06$ N/m),^{14,15} there are limitations in measuring stiff levers due to small amplitudes of oscillation and because the contribution of the principal mode to the power spectrum cannot be estimated with sufficient reliability.¹⁶

Early work proposed to determine the spring constant of levers by statically loading the tip against a pendulum of known mass and measuring the deflection.¹⁷ However, these measurements proved to be time consuming and provided results that varied by 30%–40%. Numerous researchers have used methods that deflect a lever of unknown stiffness by a precalibrated lever. The unknown lever is deflected a measurable distance by a known force derived from moving a precalibrated standard lever a known distance.^{18,19} This method maintains uncertainties associated with location of the loading, and a relatively narrow dynamic range. The accuracy achieved by such measurements is of the order of 20%.¹¹

Finally, measurements based on the physical dimensions measured by scanning electron microscopy (SEM) suffer from errors in measuring these dimensions and in substantial

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TABLE I. Comparison of the cantilever measurement methods with comments (as reported in Ref. 10).

Method	Accuracy (%)	Demerits
Added mass resonance	10	Difficult, requires micromanipulation, potentially destructive
Thermal fluctuations	10–20	Soft levers only, must analyze resonance curve shape
Scaling from resonance frequency	5–10	Requires SEM determination of dimensions and estimate of effective mass
Static deflection with added mass	15	Difficult, requires micromanipulation, potentially destructive
Response to pendulum force	30–40	Complex, time consuming
Static deflection with external standard (calibrated standard)	15–40	Requires accurate standard
Nanoindenter method	<10% depending on lever stiffness	Fast (15 min), simple, straightforward interpretation, nondestructive

variations in the physical properties of the lever itself. Errors in the measurement of the thickness scale by a factor of 3 because the spring constant varies as t^3 —thus, a 5% error in thickness leads to a 15% error in spring constant.

In the procedure described herein, we demonstrate a nondestructive method to determine accurately the cantilever spring constant by measuring the displacement of the lever due to applying a load at the point of specimen contact, regardless of the lever geometry or overall dimension. This procedure utilizes a nanoindentation module with a capacitive displacement transducer attached to a commercial AFM. The applied force and resulting displacement are measured simultaneously. While the use of a nanoindenter has been used to measure micromechanical properties, particularly hardness and elasticity, to the best of our knowledge this is the first application for determining the spring constants of cantilevers specifically for use in atomic force microscopy.^{20–22}

Table I is a comparison of the estimated (i.e., reported) accuracy of various measurement techniques.¹⁰ As may be seen, the accuracy varies from 10% to 50%. In order to avoid confusion about the meaning of the word “tip” (there are three of them), the following wording will be used: “lever end” will be used to describe the tip of the lever, that is the physical end of the lever arm(s); “probe tip” will be used to describe the tip that is used by the AFM to scan images; and “indenter tip” will be used to describe the tip used by the nanoindenter to make the measurement.

B. Elastic properties of cantilevers

The mechanical response of cantilevers may be modeled using classical mechanics that determine the bending of an elastic single-layer beam. Rectangular beams have the following dimensions that define their inherent stiffness; L , the length of the moment arm, the thickness t , and the width w . The “spring constant” of such a lever can be shown to be

$$k = \frac{Et^3w}{4L^3}, \quad (1)$$

where E is the material modulus of elasticity.^{23,24}

Microfabricated silicon-nitride cantilevers exhibit inherent thickness deviations from one lever to another and from batch to batch that when cubed, as in Eq. (1), cause considerable inaccuracies.²⁵ For instance, the spring constant of Ultralevers[®], produced by ThermoMicroscopes (Sunnyvale, CA) from (111)-orientated doped Si [$E = (1.25 \pm 0.06) \times 10^{11} \text{ Nm}^{-2}$] with an approximate thickness of $0.7 \mu\text{m}$ as determined by SEM (accuracy = $\pm 3\%$), can be determined with an uncertainty of $\pm 20\%$ (with $3\Delta t$ being the principal source of uncertainty) using Eq. (1). However, in practice, due to the formation of nonstoichiometric silicon nitride and anisotropic growth of the material system, the modulus of elasticity is uncertain, and it is difficult to determine k with confidence without direct measurement.²⁶ In fact, our results suggest that variations in k can be large, even for nominally identical levers grown next to each other on a wafer.²⁵ Such levers should have very similar physical dimensions and yet have widely differing spring constants.

The spring constant for a V-shaped lever has been analytically determined by several groups who have interpreted the parallel-beam (PBM) approximation by different means.^{27,28} The parallel-beam approximation follows the principle that the V-shaped cantilever is approximated by two rectangular cantilevers joined in parallel, thus allowing the analytical result of a rectangular cantilever to be applied to this specific case.²⁹ The corrected PBM approximation offers a more accurate approximation of the force constant in the following:

$$k_N = \left[\frac{Et^3w}{2L^3} \right] \cos \theta \left[1 + \left(\frac{4w^3}{b^3} \right) (3 \cos \theta - 2) \right]^{-1}, \quad (2)$$

where θ is the angle between the two arms of the lever, w is the width of the beam parallel to the base line, and b is the width of the “V” at the base.³⁰ This analysis assumes the load is applied at the end tip of the cantilever.

If the tip is at a distance ΔL from the lever end, a correction factor can be applied:

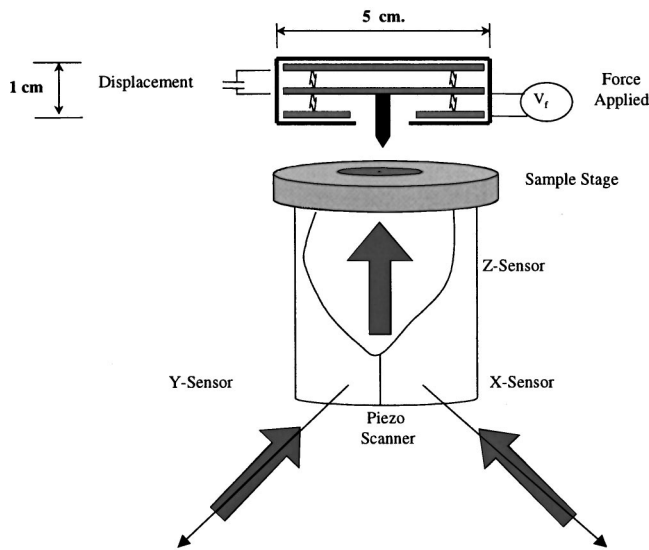


FIG. 1. Schematic of ThermoMicroscope piezoscanner and Hysitron transducer suspended on top, with the sample mounted on a stage placed on top of the piezo.

$$k_{N,\text{eff}} = k_N \left[\frac{L}{L - \Delta L} \right]^3, \quad (3)$$

where $k_{N,\text{eff}}$ is the effective spring constant of the cantilever and ΔL is the offset distance of the tip with respect to the end of the lever.¹⁴ For a typical short lever, L is of order 100 μm and ΔL is of the order of 3 μm , so the correction factor is almost 10%. Longer levers have smaller correction factors.

The important issue is that the form of the spring constant is unchanged by the geometry of the lever configuration. The spring constant k varies as the thickness cubed and the inverse of the length cubed.

C. Nanoindenter transducer

The Hysitron[®] nanoindentation transducer (Fig. 1) consists of three parallel plates forming two capacitors. The middle plate is suspended by springs between the outer two, which are rigidly fixed. The indenter tip is attached to the middle of the center plate and passes through the lower plate via a small hole. A voltage V_f is applied to the center plate and a resulting force is measured. The displacement is measured at the same time by measuring the capacitance between the center and upper plates. This simultaneous measurement of force and displacement gives the transducer the ability to give quantitative force–displacement curves. From the force–displacement curve of an indent at the end of a lever, we can calculate the force constant.

A number of assumptions are made in this measurement. First, the actual indentation made in the surface of the tip is negligible. The forces involved are small, so any indents will be small in depth compared to the deflection lengths (a few nm compared to hundreds of nm). Second, the compliance of the AFM/nanoindenter combination is negligible compared to the compliance of the lever. Both of these assumptions are reasonable. The compliance of the AFM/nanoindenter is known to be of the order of 10^{-6} m/N, while a lever's compliance is of order 1 m/N. Third, it is assumed that the

nanoindenter load is applied normal to the surface of the lever, and that the deflection of the lever is not large enough to change this angle. Since the levers are, typically, 100 μm or more in length, and deflections are, typically, less than 500 nm, changes in the angle of the load are small. Of more concern is the ability to set up the apparatus so that the load is indeed normal to the lever. In fact, the error in this angle may be as large as 5° . However, the normal component of the load will be the actual load times $\cos(5^\circ) = 0.996$, so this error will not be significant. Finally, it is assumed that the indenter tip does not slip laterally. In fact, such slips do occur for very large deflections (in excess of 500 nm) and are very noticeable as discontinuities in the force–displacement curve. The typical deflections used here are of the order of 100 nm, or less than 0.1% of the length of the lever.

D. Indenter tips

The most important and difficult task in measuring k is the alignment of the indenter tip with the probe tip. Ideally, the indenter tip presses against the lever at the point on the backside of the lever exactly above the probe tip. The difficulty arises from the lack of a clear optical path around the indenter tip as it contacts the backside of the lever. In addition, alignment must be performed in both the longitudinal and lateral directions, which requires an optical view from two perpendicular directions.

Two different tip shapes are used; a classically etched scanning tunneling microscope (STM) tip with a high-aspect ratio and a wedge-shaped tip configuration with a high-aspect ratio in one direction and a low-aspect ratio in the other. Results using both tips were to within $\pm 2\%$.

The high-aspect ratio STM tip provides excellent optical access around the tip. By using two optical views the alignment is accomplished to within a few microns in both directions quite easily. This tip may be easily “walked” across the surface using the manual controls or using the AFM scanner to apply offsets.

The wedge-shaped tip is designed to eliminate one degree of freedom, making the alignment easier and less time consuming. By orienting this chisel edge perpendicular (nominally, to within $\pm 3^\circ$) to the length of the lever, the “y” direction degree of freedom is eliminated—no matter where the lever is in the y direction, the wedge crosses the lever. Optical alignment is performed to within a few microns in the x direction and the y alignment is assumed to be correct.

For both designs, the tips are mounted with cyanoacrylate adhesive into a tip holder. The compliance of this arrangement is small compared to the compliance of the levers, and will, therefore, not affect the results. This finished tip has been viewed under an environmental scanning electron microscope (ElectroScan 2020, Phillips Electronics, The Netherlands) to insure tip geometrical accuracy (Fig. 2).

Wedge tips are made by mechanically shaping tungsten wire by hand. Hand shaping has been performed on a diamond wheel with the tip mounted in a hand-held positioning device allowing for a light polish on the wheel (Fig. 3). The resulting tip is uniform, with an edge length of 400 μm proving quite satisfactory for measuring cantilevers.

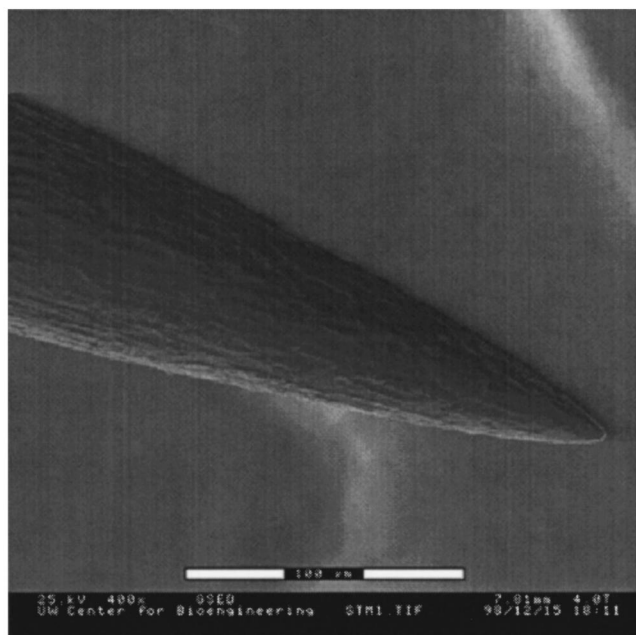


FIG. 2. STM etched tip.

The main concern with the chisel end is that we must assure it makes contact with the lever in at least two places. If there is only one, the tip will rotate until a second point is in contact. The issue is how far apart those points are and whether those points change during an indent altering the data. If points are close together, as is potentially the case of a STM tip, this may be treated as a single point because the torque applied to the lever will be essentially the same from each point and will not affect the data. If the points are far apart, as is potentially the case for the chisel, the tip will rotate during initial contact with the first point until the second point of contact is made. From the second event, data will be consistent and may be treated as an ideal contact. In practice, the tip is held in contact with a minimum set-point

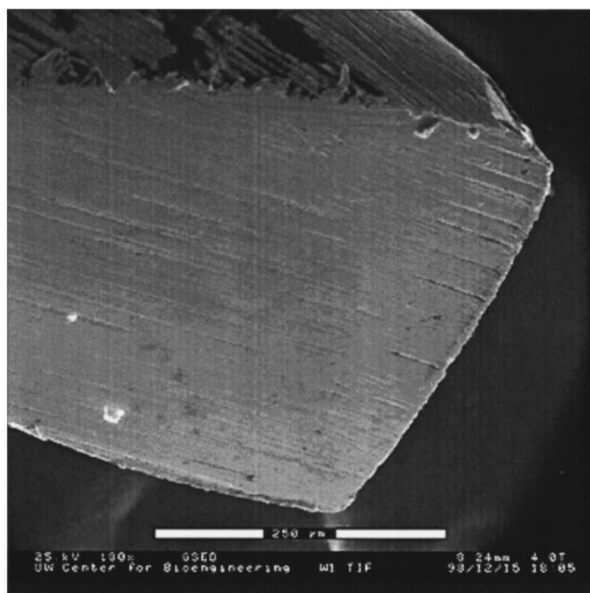


FIG. 3. Hand-machined chisel tip produced from tungsten wire.

force of $3 \mu\text{N}$, thus both points are in contact at all times. Finally, if the two points of contact are somewhere in between these two extremes and the tip/lever contact varies throughout the indent, such as in the case of a rounded chisel tip where the point of contact may vary by many microns, it can lead to poor data. However, using the two tips described, we obtained measurements that were consistent with a single-point contact, and in the rare occasion that we obtained suspect data, it was due to operator error and was rejected.

II. SOURCES OF ERROR

A. Random errors

Random errors in the measurements arise primarily from two sources: electrical and vibration. In both cases, the effect is to add a background noise to the signal measured by the transducer. The main effect of this noise is to increase the minimum force constant that can be measured. As a result of this noise, the smallest k that can be measured reliably is roughly 1 N/m .

Experiments were performed in two locations. In each case the AFM used was a ThermoMicroscopes AutoProbe CP (Sunnyvale, CA).³¹ At ESR, Inc., where a Minus K[®] vibration isolation table was utilized, the largest source of noise was the electronic noise on the signal, primarily 60, 120, and 180 Hz. At the University of Washington, with no isolation table, the primary noise source was low-frequency vibrations. These noise levels could not be reduced in spite of several attempts to eliminate ground loops, etc. The noise level was roughly 200 pA rms as measured at the Thermo Microscope[®] CP (current) or about 20 mV as measured by the Hysitron data acquisition system.

B. Instrumentation errors

There are three instrumentation constants used in the measurements: the electrostatic force constant (EFC), the load scale factor (LSF), and the displacement scale factor (DSF). In order to obtain accurate and precise measurements of the force constant of a lever, all three constants must be measured. Typically, the electrostatic force constant varies slightly with time, while the other two do not change.

C. Electrostatic force constant

The electrostatic force constant, which compensates for the mechanical resistance of the springs imposed on the suspended plate, is the primary source of systematic error in these measurements. The Hysitron[®] software uses this constant to calculate the electric field for a given voltage and displacement. However, the EFC, whose units are $\mu\text{N/V}^2$, is easily measured and can be estimated accurately enough that errors of only a few percent are possible. The EFC must be measured periodically to ensure it has not drifted and it must also be measured any time a change is made to the transducer such as after installing a new tip.

The EFC is measured by performing an “indent” when the tip is far from the surface; a so-called “air” indent. During this indent the central plate moves freely, with the resis-

tance to motion of the plate coming only from the stiffness of the springs holding it in position. Thus, for any given voltage, the plate should move only as far as the force is able to deflect the springs. The Hysitron[®] software includes a “spring force compensation” term that subtracts this force. In a real indent, the important quantity is the force due to the resistance of the substrate to the tip—the resistance of the spring is of no interest and is, therefore, subtracted out.

Thus, an air indent should show a load–displacement curve which is flat—zero additional resistance for all displacements. By performing an air indent, the user may look at the load–displacement curve, and assuming the mechanical resistance of the plate suspension is slightly out of adjustment, one varies the EFC until the resultant curve is flat, insuring correct calibration. In practice, at large displacements some small deviation from linear is observed. This is due to nonlinear electrostatic effects—the software assumes that the parallel-plate approximation is valid, and this appears to be true for displacements of up to 200 nm. When measuring a lever force constant the user must, therefore, make sure that the displacement is limited to less than 200 nm.

D. Load scale factor

The load scale factor is a second transducer constant used to convert the electric field into a force. This factor, whose units are mV/N, can be calibrated by comparing the force measured by the transducer (Newtons) with the force measured by a second instrument (in this case, we chose to measure the load using a very sensitive balance that has a nominal accuracy of 0.001 mg). To compare the same force measured by both instruments, the difference between the two is determined. Therefore, the ratio of the transducer measurement to the load measurement is the gravitational constant g .

To perform this calibration, we built a “bridge” structure to support the Hysitron[®] transducer above the active surface of the balance. (This aluminum structure was mounted above the sample stage to support the Hysitron[®] transducer depicted in Fig. 1.) This bridge contained 80 pitch thumb screws to permit a slow, controlled approach, with a resolution of a few microns. The approach was monitored using a long focal length optical microscope.

It was necessary to apply a “preload” to the balance to ensure the tip was in contact. In these measurements the preload was adjusted by gross amounts three times, with no visible change in results. The preload varied between 59 and 345 mN, as measured by the balance. Also, the preload varied slightly with time, due primarily to the compliance of the transducer support structure. For each measurement, the preload was subtracted from the final measurement to give the actual force applied. The mechanical stability of the system produced a random drift of order 492 μ N. We believe this was primarily due to changes in the compliance of the balance and/or the bridge support structure.

The applied load (from the Hysitron[®]) and resultant approach weight as measured by the balance are related by the gravitational acceleration g . Relating these two measure-

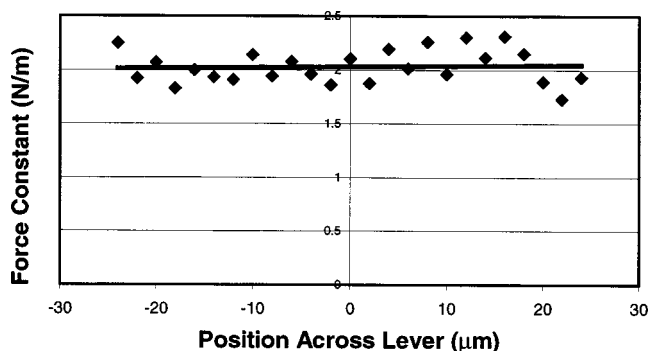


FIG. 4. Measurements across Ultralever A 25 μ m from the tip position.

ments and taking into account the noise in the system and random drift of the support, the uncertainty in our measurement is $\pm 5\%$. The effect of this uncertainty is that the values for k that we measure may have a systematic error as large as 5% (one sigma). Clearly, a better method for calibrating the forces would help reduce the total systematic errors in measuring k .

E. Displacement scale factor

Displacement is measured directly by the transducer using the variation in capacitance between the top two plates. The Hysitron[®] software makes the assumption that the two plates can be approximated by an ideal parallel-plate capacitor. This works well for displacements below 500 nm, but above that it becomes increasingly poor. The software uses the displacement scale factor to calculate the displacement based on the capacitance.

We were unable to measure the DSF factor for accuracy and used the value provided by Hysitron[®]. Our measurements may, therefore, contain a systematic error due to errors in the DSF. Hysitron[®] claims to have an accuracy of 3%–4% for this value.

F. Misalignment errors

Misalignment of the indenter tip on the back of the cantilever will cause a systematic error from two sources: longitudinal errors will result in errors in k because the stiffness depends on L^{-3} ; and lateral errors will cause additional

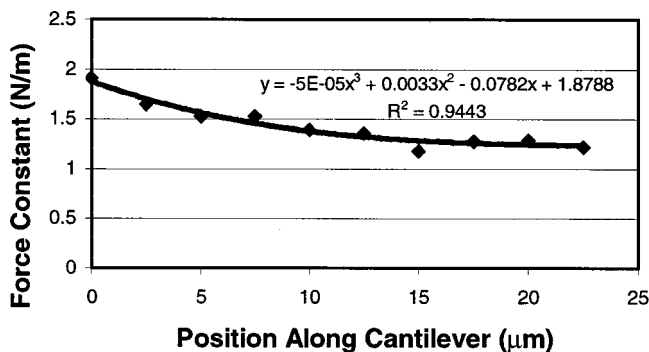


FIG. 5. Sample calibration of Ultralever noncontact cantilever.

TABLE II. Summary of transverse measurements performed on Ultralever A and B levers.

Lever	Lever type	k (N/m)	Distance from end of tip (μm)	Lateral variation (N/m per μm)
Ultralever	Noncontact: A	2.0	25	0.003
Ultralever	Noncontact: B	4.0	40	0.012
Ultralever	Contact: A	1.1	5	0.00
Ultralever	Contact: B	1.3	8	0.007

rotational motion of the end of the lever that will appear as a lowered stiffness value.

We can estimate how much error in k a lateral misalignment will introduce if we know how k varies across the lever. This variation was measured by making closely spaced indents and determining the “edge” of the lever by noting at which point the tip fell off. The tip was moved 1 or 2 μm per step using the AFM scanner tube. This scanner, from ThermoMicroscopes, includes a closed-loop scanner nonlinearity correction called SCANMASTER[®], which eliminates scanner nonlinearity, hysteresis, and creep, and is accurate to approximately 10 nm.

We measured k across several levers (transverse to the lengthwise direction) with a STM tip in an attempt to determine the variation in stiffness as a function of position with respect to the centerline. A sample series of measurements on one lever is presented in Fig. 4. The force constant variation is up to 18% at the extreme off-center, as one would expect. More importantly is that within 10 μm of the center line, a position easily obtainable using optical alignment, the variation is less than 7%.

In summary, our data show that the variation is small and to all intents and purposes can be ignored. The summary of a series of measurements we have performed is presented in Table II. It would appear that lateral alignment is relatively unimportant, introducing small changes of the order of a percent at worst. These results indicate that lateral alignment is not a critical task to achieving accurate measurements. This allows for a more robust and rapid test procedure.

The variation of k along the lever is also important in determining the accuracy with which the indenter tip must be aligned. One can make theoretical estimates about the variation based on the assumption that the variation of k with length goes as L^{-3} . For a lever of length 100 μm , a variation in L of 1 μm leads to a change of $(100/101)^3$ or as expressed in percent, about 2.9% per μm . For a Park Ultralever Tip A with a nominal length of 180 μm and a measured stiffness of 1.1 N/m, we measure a variation of 0.027 N/m per μm or about 2.5% per μm .

The effect of longitudinal misalignment is clearly more important than lateral misalignment, especially for short levers. Using a good optical microscope and careful illumination, the lever tip appears as a small speck of light. Using the AFM scanner to move the lever, one can move the indenter tip to within two or three μm of the tip. The limiting factor is the optical microscope.

A sample series of measurements may be seen in Fig. 5. The force constant is plotted versus the position on the lever. In this case, the cantilever end is approximately 22.5 μm

from the initial measurement, although the actual tip typically is positioned 3 μm from the tip of the cantilever.

III. PROCEDURE

A. Chip holder

A chip holder design must allow sufficient optical and physical access to the lever. Cantilevers may be mounted in any fixture for this technique, including the standard Park AFM unmounted tip holder. We have also produced special holders from machined copper stock with a notched groove to hold the fragile cantilevers. The notched block captures the cantilever with a piece of 0.127 mm stainless-steel shim stock brazed onto the block. In addition, Park AFM pre-mounted levers on Macor ceramic blanks or levers glued directly to AFM pucks may be tested. The orientation of the chip should be such that the tip faces down, so the indenter will make contact with the backside of the lever.

B. Optics

It is necessary to have two perpendicular views of the lever and indenter tip unless a chisel-shaped tip is used. This permits accurate alignment on the backside of the lever in both X and Y . For alignment in the X and Y directions, we used a long focal length optical microscope with a resolution of a few microns. We have combined this optical microscope with a small first silvered mirror at 45° . The main view was of the lever side on, with the tip approaching from above. The view in the reflected mirror was of the lever, end on.

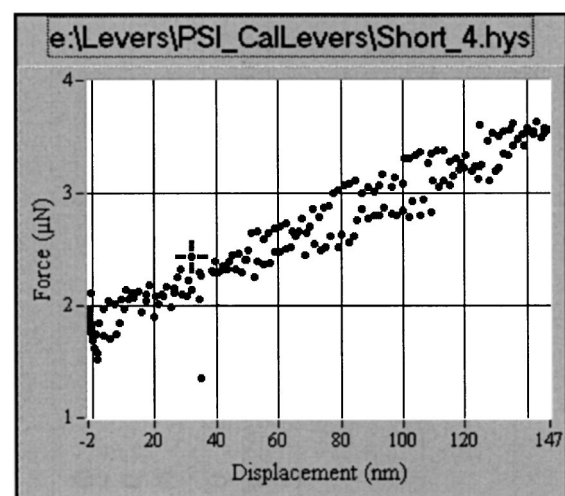


FIG. 6. Typical force-displacement curve. Maximum displacement: 147 μm .

TABLE III. Comparison of the measurement of calibrated levers with the nanoindenter technique.

Lever	Resonant freq. (N/m)	Nanoindenter results (N/m)
Middle	1.3 ± 0.1	1.2 ± 0.2
Short	11.7 ± 0.2	11.9 ± 0.3

C. Methodology

The following methodology was used to make the measurements. A key feature of this process was a periodic check for variation in the electrostatic force constant, which was the major source of systematic error.

Using the Z stepper motor, move the tip to within 1 mm of the height of the lever. Use the X and Y screws on the head to move the indenter tip directly above the lever. Use both optical views to ensure accurate alignment. Align the STM tip over the end of the lever approaching to within 5 μm of the end of the lever.

Perform a 25 μN indent in air. The data points should form a flat line parallel to the X axis. Determine the gradient, which should be less than 0.05 N/m. If it is not within this range, use the Hysitron[®] software to change the EFC by a small value (approximately 0.0005). Determine the gradient again and repeat until it is less than 0.05 N/m.

Using the stepper motor while watching in the optical views, approach the tip to within less than 100 μm of the lever. Carefully align the tip with the lever, using the X and Y screws and if finer control is needed, using the X and Y offsets in the AFM software.

Confirm that the AFM is correctly measuring the current by comparing the readout on the Hysitron[®] controller with the current measured by a digital volt meter in the ThermoMicroscope ProScan[®] software. Select the approach button and watch the optical view to ensure the tip is not off center to the cantilever.

Next, a maximum load of 25 μN is entered. Perform an indent and view the load versus displacement curve. It should be a straight line without curvature. The presence of steps are due to slippage while curvature may be due to nonlinearities in the transducer that occur for indents larger than approximately 150 nm. In either case, reduce the indent load and repeat. Using the AFM controls, move the tip towards the end of the lever by 5 μm . Repeat the indent. Continue moving 5 μm until the tip reaches the end of the lever. At this point, retract the tip 100 μm .

Repeat the air indent to check the EFC. This check must be made after changing tips, and after each lever, to insure the setting has not drifted.

IV. MEASUREMENTS

Measurements were made on a variety of levers. First, calibrated levers manufactured by ThermoMicroscopes were measured to insure the accuracy of the technique. Once a confidence level was reached in the accuracy of the technique, cantilevers from several manufacturers were measured including ThermoMicroscope (Microlevers and Ultralevers, Sunnyvale, CA), Nanosensors (tipless cantilevers, Wetzlar-Blankenfeld, Germany), and NT-MDT (NSCS 12 and CSCS 12, Moscow, Russia).

A. Comparison with ThermoMicroscope calibrated levers

ThermoMicroscope produce a series of force constant calibration levers that may be used to calibrate cantilevers. The calibration levers are a group of three levers of different length manufactured on the same chip. It is assumed that all three levers have essentially identical thickness and Young's modulus. By measuring the resonant frequency of all three, and measuring the widths and lengths, it is possible to solve for the thickness, and hence, the stiffness of each lever. We obtained a set of these levers that were calibrated by a collaborating research group using this method. Subsequently, these levers were measured directly using the method described herein. The nanoindenter results agreed with the resonance results and were well within experimental error, as depicted by Table III.

It is notable that the time taken to measure the stiffness with the nanoindenter was of the order of 20 min to do both, and this included repeating the measurements 15 times to ensure statistical accuracy and ensure that the measurements were made at the end of the lever.

B. Example data for several types of lever

A typical load–displacement curve is shown in Fig. 6. It is linear over the whole range and has a root-mean-square noise of approximately 0.25 μN . This noise is due primarily to vibrations (this measurement was taken at the University of Washington without a vibration isolation table).

Our data indicate that the variation along the tip depends on the lever, as would be expected. The variation is approxi-

TABLE IV. Comparison of the measured spring constants of several cantilevers to their nominal spring constant values.

Lever	Lever type	Nominal length (μm)	Nominal k (N/m)	Measured k (N/m)	Variation (N/m per μm)	Variation (% per μm)
Ultralever	A: noncontact	180	1.9	2.0	0.032	1.6
Ultralever	A: noncontact	180	1.9	1.2	0.027	2.5
Ultralever	B: noncontact	180	2.8	2.1	0.036	1.8
Ultralever	B: noncontact	180	2.8	1.6	0.043	2.8
Microlever	Lever F	85	0.5	1.6	0.011	0.7
Nanoprobe	Tipless	225	1.2–5.5	1.5	0.029	2.0
CSCS21	B: contact	110	3.0 ± 1	5.1	0.053	1.9
NSCS12	B: noncontact	90	6 ± 1	4.3	0.071	1.6

mately cubic over large ranges, however, it can be approximated by a linear variation near the tip (within 5 or 10 μm). A table of data from numerous levers is presented in Table IV. In a subsequent publication, we report measurements of several levers in detail.²⁵

C. Error analysis

The total error of the measurement technique results from the cumulative effect of several sources outlined in Sec. II. Measurement comparisons with calibrated levers that had been independently characterized were accurate to within 7.6% for a force constant of 1.3 N/m and to 1.7% of a lever with a force constant of 11.7 N/m. The precision (we performed a group of six measurements of each lever) of the measurements was 4.5% for the weaker levers and 8.9% for the stiffer levers. Thus, we believe that the Hysitron[®] nanoindenter may be utilized to calibrate scanning probe microscopy cantilevers with an accuracy of better than 10% to a precision of better than 10%.

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³¹ThermoMicroscopes[®] CP: Two different CPs were used with identical results.