Calibration of the torsional spring constant and the lateral photodiode response of frictional force microscopes

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We present a direct one-step technique to measure the torsional spring constant of cantilevers used for lateral or friction measurements with the atomic force microscope. The method simultaneously calibrates the photodiode response to the angular deflection of the cantilever. It does not rely upon any approximate theory for friction, nor upon any simplified model cantilever geometry or elasticity. The technique is verified by comparison with the calculated spring constant and with an independent measurement of the angle calibration. This nondestructive calibration may be performed with any type of cantilever, and the friction may subsequently be measured with any type of substrate or probe. © *2000 American Institute of Physics.* [S0034-6748(00)01507-0]

I. INTRODUCTION

The quantitative measurement of friction with an atomic force microscope (AFM) or friction force microscope (FFM), in general involves two steps: the calibration of the lateral photodiode response to convert the measured volts to the angle of twist of the cantilever, and the measurement of the angular spring constant of the cantilever, which converts angle to torque.

Most calibration techniques calculate the spring constant rather than measure it.[1–5] For the common V-shaped cantilever, finite-element analysis or algebraic formulas for the torsional spring constant have been given.[6] Such calculations are approximate in the sense that they use simplified models of the cantilever (e.g., ideal geometry, bulk elastic properties, neglect of coatings, uniform thickness). Toikka *et al.*[7] attempted to measure the torsional spring constant from the twist due to the gravitational moment exerted on a glass fiber glued perpendicular to the cantilever. The value of the spring constant that they obtained is several orders of magnitude smaller than their calculated value. Since the cantilever twist due to gravity may readily be shown to be negligible, it appears that they were in fact measuring the saturation of the photodiode rather than any change in twist angle. There currently exists only one method for the direct measurement of the torsional spring constant,[8] which is discussed later. This is in contrast to the normal or vertical spring constant where a number of direct measurement techniques exist (e.g., resonance, gravitational).

A number of methods have been proposed for calibrating the photodiode response to the twist angle. Perhaps the most direct is the method of Meurk *et al.*,[9] in which the stepper motor is used to tilt a mirrored substrate, and the output voltage is measured as a function of angle. Alternatively, Liu *et al.*[2] proposed that the sensitivity of the lateral photodiode was proportional to the sensitivity of the vertical photodiode, which is readily measured, and the ratio of the total lateral signal to the total vertical signal. Liu *et al.*[3] modeled the beam path in the AFM and calculated the lateral sensitivity from geometrical considerations. A number of workers have attempted to obtain the calibration factor from the initial slope of a fraction loop.[1,7] Assuming that the tip is pinned to the substrate, and that the lateral piezo movement has been accurately calibrated, then the angle of twist can be obtained. Nonlinearity in the lateral piezo movement, which can be of the order of 30%,[8] or any slippage or deformation of the tip renders this method inaccurate. Accordingly a number of workers have attempted to use simplified models of friction and elastic deformation to overcome the latter restriction.[4,5] Obviously direct calibration methods such as that of Meurk *et al.*[9] are preferable to those that neglect or approximate such artifacts.

In addition to the earlier two-step calibration procedures, a number of alternative methods for measuring friction or for calibration have been proposed. Ruan and Bhushan[10] suggested that friction could be measured in the direction parallel to the long axis of the cantilever, and that in essence the vertical spring constant times the vertical piezo movement necessary to hold the cantilever deflection constant gave the frictional force. The friction measured in the normal direction with this method has been used to calibrate lateral friction measurements.[11] Unfortunately, this method neglects the bending moment of the cantilever, which has been shown to affect the measured friction force[5,12] and so it cannot be considered a quantitative method. The most commonly accepted method in current use is that of Ogletree *et al.*[13] Here friction loops are required as a function of applied load on substrates with two well-defined slopes. The limitations of the method are the requirement of multiple measurements, the necessity of electronic feedback correction for crosstalk (since the lateral signal can be less than 2% of the vertical signal), the need for a special substrate, the restriction to a cantilever with a sharp tip, and the use of the Johnson–Kendal–Roberts (JKR) approximation (that friction is proportional to the load plus the adhesion). Most recently, Bogdanovic *et al.*[8] directly calibrated the cantilever by pushing it against a protuberance

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off-set from the cantilever axis. The torsion is obtained from the measured force (vertical deflection) and the lateral off-set, which is obtained from the calibrated lateral piezo movement. The calibration is completed by using the method of Meurk *et al.*[9] to calibrate the photodiode response to the twist angle. This method has several advantages over that of Ogletree *et al.*:[13] it can be applied to any cantilever, no special substrate is required, and no theoretical approximation is invoked. Its disadvantages are that the lateral scanner has to be calibrated and corrected for nonlinearities, and that the zero of the offset has to be known, which requires fitting to multiple measurements.

In this article we present a method to determine experimentally the friction force from the lateral voltage signal. Our calibration procedure requires the simultaneous measurement of the vertical and lateral deflection of the AFM cantilever due to the force on a lever attached to the cantilever. Toikka *et al.*[7] previously employed a similar lever arrangement, and Bogdanovic *et al.*[8] also monitored both photodiode signals. The novelty in our work is the formula that we derive for obtaining the calibration factor for converting the lateral photodiode signal to an applied torque. Compared to the method of Bogdanovic *et al.*,[8] the use of a lever improves the signal-to-noise ratio and minimizes the effect of possible crosstalk on the lateral signal. Importantly, this method can be applied to cantilevers of any size and shape and those with or without tips. This calibration factor may be used subsequently to measure quantitatively the friction force between any substrate and any tip or attached colloid probe.

By obtaining directly the friction calibration factor, we bypass the two-step procedure mentioned in the opening paragraph. Our method does additionally yield the sensitivity factor of the photodiode for angular deflection and the cantilever twist spring constant. The sensitivity factors are not directly required for a friction measurement, but they serve as a test and as a guide to the accuracy of the method. In particular, here we compare the angular sensitivity measured with our method with that obtained by the method of Meurk and co-workers.[9] We also compare our measured torsional spring constant with that calculated from the dimensions of the V-shaped cantilever using the approximation of Neumeister and Ducker.[6] As a final test of the method, results for several independently measured cantilevers show a reproducibility of better than 10% and no dependence upon the length of the attached lever.

II. EXPERIMENT

All AFM experiments were performed using a Nanoscope III instrument (Digital Instruments, USA), the cantilevers used were commercial *long wide* triangular cantilevers (length=193.2 μm, width=36.8 μm) also sourced from Digital Instruments. In a routine force measurement the substrate exerts a vertical force F on the cantilever such that $F = k_v \Delta z^0$, where k_v is the vertical spring constant and Δz^0 is the vertical deflection (see Fig. 1). We measured k_v using the method of Cleveland *et al.*[14] The vertical detector sensitivity, α, is the slope of the constant compliance region of the force-distance curve

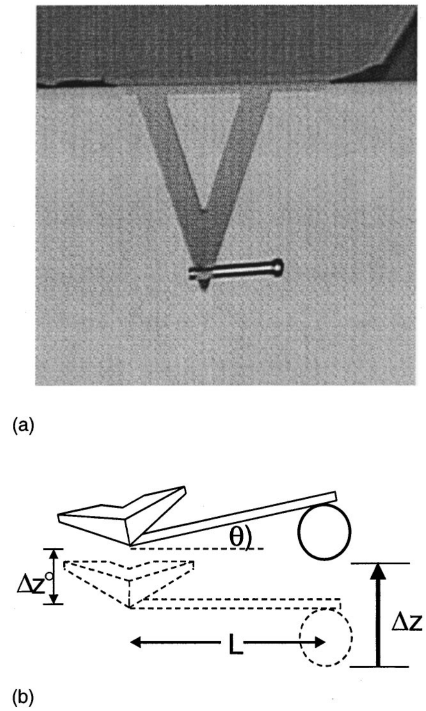

FIG. 1. (a) Optical microscope image of AFM cantilever with attached lever assembly, consisting of a glass fiber of length 100 μm and a silica sphere of radius 5 μm. (b) Schematic showing the vertical deflection, Δz^0, and the angle of twist, θ, of the cantilever with an attached lever of length L due to an applied torque due to a piezo movement of Δz.

$$\alpha = \frac{\Delta z^0}{\Delta V_{\rm vert}} (\mathrm{m/V}), \tag{1}$$

where $\Delta V_{\rm vert}$ is the change in the vertical voltage signal. It is worthwhile to note that for experiments with the same cantilever (i.e., remounted), and with different cantilevers from the same batch, α varies by less than 10%.[15]

In Fig. 1(a) we show an AFM cantilever with a lever of length L attached using the method of Ducker *et al.*[16] and, in (b), a schematic showing the normal deflection and twist of the cantilever due to an applied torque. The bending of the glass fiber is negligible.[17] We have attached a sphere to the end of the lever to ensure that the distance from the cantilever to the point of contact is accurately known. However, as we show later, levers without spheres can be used equally well, provided that contact is only with the free end of the lever. The vertical and lateral deflection of the cantilever due to the applied force, F, and torque, $\tau = FL$, result in a change in vertical, $\Delta V_{\rm vert}$, and lateral, $\Delta V_{\rm lat}$, voltage signal, as shown in Fig. 2. The signals are recorded simultaneously and, from Fig. 2, one can see that a change in the lateral deflection corresponds directly to a change in the vertical deflection.[18] Calibration factors for both vertical and lateral deflections can be measured from the constant compliant slopes of the force-distance curves

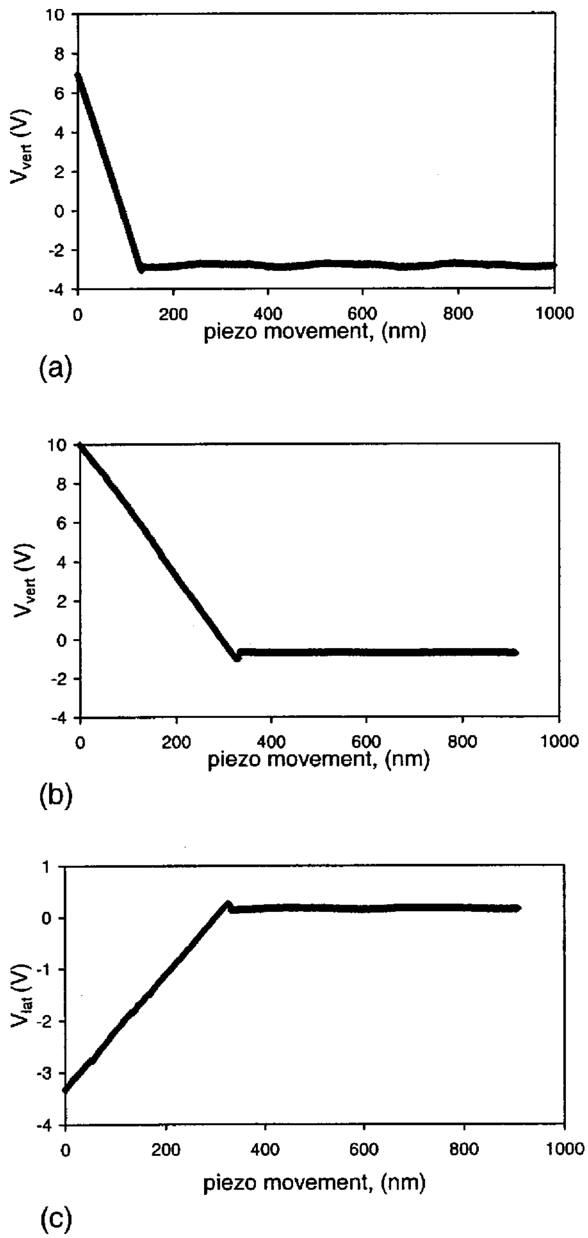

FIG. 2. The vertical deflection voltage signal as a function of piezo movement (a) with and (b) without an attached lever, and (c) the lateral voltage deflection signal with an attached lever. For clarity, only the inward trace is shown, the outward traces show similar trends.

$$\alpha_L = \frac{\Delta z}{\Delta V_{\text{vert}}} (\text{m/V}), \tag{2}$$

$$\beta_L = \frac{\Delta z}{\Delta V_{\text{lat}}} (\text{m/V}), \tag{3}$$

where α_L and β_L are the respective calibration factors for the vertical and lateral deflections with a lever attached, and Δz is the change in the piezo extension (see Fig. 1). Note that $\Delta z \neq \Delta z^0$; the former is the vertical movement of the lever tip in contact with the substrate, whereas the latter is the vertical deflection of the cantilever, consequently $\alpha \neq \alpha_L$.

We now give our central result for the calibration factor γ, that converts a lateral voltage to an applied torque

$$\tau = \gamma \Delta V_{\text{lat}}. \tag{4}$$

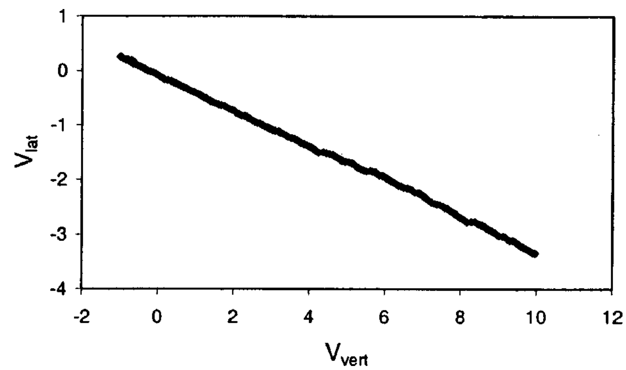

FIG. 3. The linear relationship between the lateral and vertical deflection signal. Note that this is observed over the entire piezo movement and not just in the constant compliance regions.

Using in turn the equations given earlier to express the torque in terms of the force, the force in terms of the vertical cantilever deflection, and the cantilever deflection in terms of the vertical voltage, this is

$$\gamma = \alpha k_v L \frac{\Delta V_{\text{vert}}}{\Delta V_{\text{lat}}} (\text{N m/V}). \tag{5}$$

In Fig. 3 V_{lat} is plotted against V_{vert}. It is important to notice that the relationship between the two signals is linear over the entire scan size and not just in the constant compliance regions. This confirms both the linear relationship between torque and force and also the linear response of the two photodiodes. The calibration factor γ is obtained from the slope of this line.

One can also express γ in terms of the measured vertical and lateral calibration factors

$$\gamma = \beta_L \frac{\alpha}{\alpha_L} k_v L (\text{N m/V}). \tag{6}$$

In practical terms this alternate route to γ is less accurate than by means of Eq. (5) because it relies upon the identification of the linear constant compliance regime and upon accurately finding the slopes there. Any nonlinearity in the piezo will contribute to the errors in the slope determination. Such problems do not arise in the first method, which is independent of the driving piezo. Consequently, the real use of the parameters α_L and β_L is to test the method against independent measurements of the torsional spring constant and the lateral diode sensitivity (see later).

It is now a simple matter to convert the lateral deflection signal obtained in a friction loop to a force. (Note the difference between the lateral calibration described earlier and an actual friction measurement described in this paragraph; no lever is attached to the cantilever in an actual friction experiment.) The lateral deflection signal, V_{FFM}, is taken as half the voltage difference between the trace and retrace curves in a typical friction experiment. The frictional force F_f acting on the cantilever tip or probe of height h exerts a torque $\tau = hF_f$. Accordingly, from Eq. (4), it is related to the lateral deflection signal by

$$F_f = \gamma V_{\text{FFM}} / h. \tag{7}$$

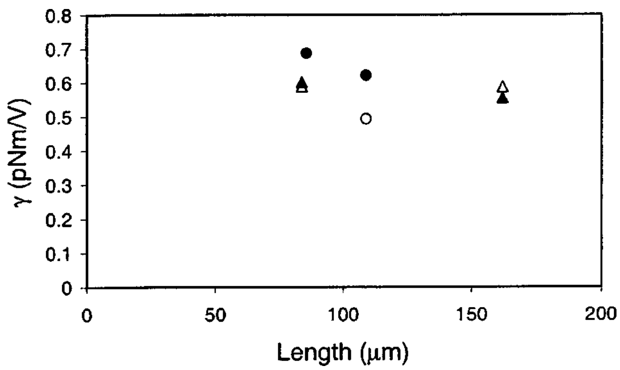

FIG. 4. The calibration factor γ obtained for cantilevers from the same batch with levers of different lengths with (●) and without (▲) spheres attached to the end. The filled symbols correspond to inward trace and the open symbols outward trace. The average value of γ obtained is 0.60 pN m/V with a standard deviation of 0.06.

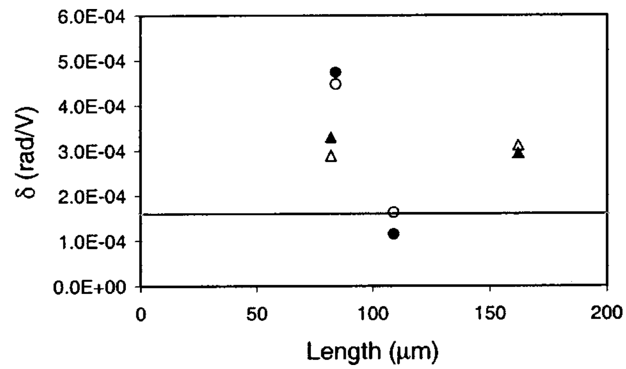

FIG. 5. Lateral photodiode sensitivity, δ, calculated from Eq. (8) using the calibration factor α_L for the four cantilevers. The solid line is the measure lateral photodiode sensitivity (1.69×10^{-4} rad/V).

The factor γ is the same as the one measured with an attached lever provided that the cantilever is identical to the one used in the calibration and that the laser and photodiode are positioned as in the original calibration. The latter can be ensured by maximizing the total vertical photodiode signal and making sure the lateral signal is zero during a normal force measurement.

Values for γ obtained for cantilevers from the same batch with levers of different lengths are plotted in Fig. 4. There is good agreement between all four cantilevers showing that γ is independent of the length of the lever. This also shows that the cantilevers can be mounted and the laser aligned consistently. The value we obtain is $\gamma=0.60\pm0.06$ pN m/V. To guarantee that the lever comes into contact with the substrate only at its tip, in two cases we attached a sphere to the end of the lever (lever lengths 84 and 109 μm). It can be seen that there is no significant difference in the value of γ between the results from those levers with sphere and those without. This confirms that only the free end of the bare lever was in contact with the substrate. If this were not the case, the value of L used in the calculations, which was the full length of the lever, would not yield consistent results and, in addition, the relationship between the lateral and vertical voltages would not have been linear over the whole regime. The gravitational pull on the lever gives a deflection of 2×10^{-3} nm which is negligible. The reason that it is only the tip of the lever that is in contact with the substrate is a consequence of the gluing procedure that creates a slight angle of the lever with respect with the cantilever. This reduces the need to attach a sphere onto the lever, which is obviously advantageous. After the calibration the lever can be removed and the cantilever reused in an actual friction force measurement. It may be seen from the data, with one exception,[19] there is also good agreement between the calibrations performed on the inward and outward runs. Although in principle, the calibration method works with any substrate, we found that best results were obtained when adhesion between the lever tip (or attached sphere) and the substrate was minimized. The results presented here were for a silica lever (or sphere) and a silica substrate, which, due to the double layer repulsion, showed minimal adhesion in ultrahigh purity water at natural pH.

III. VERIFICATION

The difference between the vertical movement of the tip of the lever and the vertical movement of the cantilever itself, divided by the lever length, gives the change in angular deflection of the cantilever, $\Delta\theta$. That is,

$$\Delta\theta=\frac{(\Delta z-\alpha\Delta V_{\mathrm{vert}})}{L}=\frac{\Delta z(1-\alpha/\alpha_L)}{L}. \tag{8}$$

Hence, the factor

$$\delta\equiv\frac{\Delta\theta}{\Delta V_{\mathrm{lat}}}=\frac{\beta_L(1-\alpha/\alpha_L)}{L}(\mathrm{rad/V}) \tag{9}$$

will convert any measured change in lateral deflection signal to angular deflection (in radians). The lateral photodiode sensitivity may also be measured by tilting a mirrored substrate using the stepper motor and monitoring the charge in lateral voltage signal.[9] We found a linear relationship between tilt angle and the lateral voltage signal and from the slope, the lateral sensitivity was 1.69×10^{-4} rad/V. In Fig. 5 this is compared to that calculated from Eq. (9) using the calibration factors measured for the four cantilevers. The calculation of δ uses values of both the vertical and lateral compliance slopes, which rely on the piezo calibration. It is

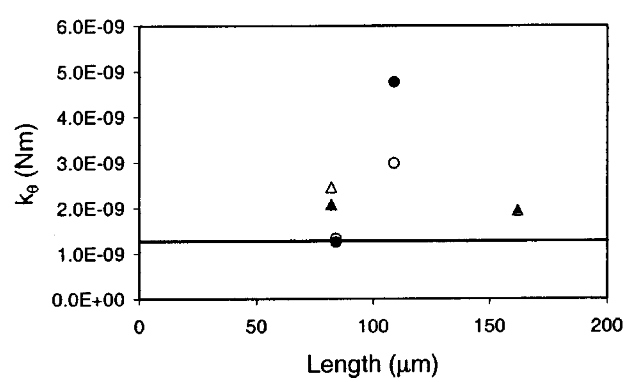

FIG. 6. Torsional spring constant, k_θ, calculated for the four cantilevers using Eq. (10). The solid line is the value calculated from the method of Neumeister and Ducker (see Ref. 6).

probable that the scatter seen in values for δ is due to the previously mentioned nonlinearity in the piezo.

The angular deflection is related to the torque via the torsional spring constant k_θ, $\tau = k_\theta \Delta\theta$. The spring constant is given by

$$k_\theta = \frac{k_v L^2}{(\alpha_L/\alpha - 1)}. \tag{10}$$

In Fig. 6, we compare our measured values of k_θ [using Eq. (10)] to that calculated from the analytic approximation of Neumeister and Ducker,[6] using the measured dimensions of the cantilever and the measured normal spring constant $k_v = 0.09$ N/m to estimate Young's modulus and the thickness. As mentioned earlier, the level of scatter in the data presented in Fig. 6 is probably due to the piezo nonlinearity; however, it may accurately reflect the variability in k_θ between different cantilevers within the same batch.

ACKNOWLEDGMENT

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[15] The sensitivity of the piezo used in our experiments was found to vary as a function of its extension. This nonlinearity, which we characterized by interference measurements, causes the slope of the compliance regions to vary with the amount of piezo extension (i.e., the nominal drive distance differs from the actual drive distance by an amount that depends upon the starting position of the drive). To minimize this error it is best to conduct all experiments and calibrations at the same piezo extension when possible.
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[17] The authors have calculated the spring constant of the glass fiber to be 1.4×10^4 N/m which is much stiffer than the cantilever vertical spring constant which is $k_{\mathrm{vert}} \approx 0.1$ N/m. Similarly, the elastic constant conjugate to the bending moment of the fiber is 1.06×10^4 N/m, which is much stiffer than the torsional spring constant of the cantilever, $k_\theta = 2\times10^{-9}$ N/m.
[18] The lateral voltage signal should be monitored even in a normal force measurement to confirm that the probe is attached on the axis and that the cantilever is not twisting.
[19] For the $L = 109$ μm case, there was an adhesion between the lever and the substrate, which results in different slopes for ΔV_{vert} and ΔV_{lat} for the inward and outward runs. The average of the two runs gives an accurate value for γ.