Calibration of lateral force measurements in atomic force microscopy with a piezoresistive force sensor

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We present here a method to calibrate the lateral force in the atomic force microscope. This method makes use of an accurately calibrated force sensor composed of a tipless piezoresistive cantilever and corresponding signal amplifying and processing electronics. Two ways of force loading with different loading points were compared by scanning the top and side edges of the piezoresistive cantilever. Conversion factors between the lateral force and photodiode signal using three types of atomic force microscope cantilevers with rectangular geometries (normal spring constants from 0.092 to 1.24 N/m and lateral stiffness from 10.34 to 101.06 N/m) were measured in experiments using the proposed method. When used properly, this method calibrates the conversion factors that are accurate to ±12.4% or better. This standard has less error than the commonly used method based on the cantilever’s beam mechanics. Methods such of this allow accurate and direct conversion between lateral forces and photodiode signals without any knowledge of the cantilevers and the laser measuring system. © 2008 American Institute of Physics. [DOI: 10.1063/1.2894209]

INTRODUCTION

Techniques for the reliable and precise calibration of atomic force microscopes (AFM) have been significant issues since AFM was developed more than two decades ago.1 There are two categories of AFM calibration: normal force calibration and lateral force calibration. The quantitative determination of absolute values of normal and lateral force conversion factors generally involves two steps: the calibration of photodiode responses and the measurement of cantilever spring constants. The spring constants can be calculated from the geometric and physical properties of the cantilevers or modeled by finite-element analysis. However, these methods are approximate as ideal models of atomic fluctuations.10 More recently still, several methods have been reported for the calibration of the normal constant.11–15

A newly reported “piezosensor” uses a piezoresistive cantilever as an active force sensor to calibrate the normal spring constants of the AFM cantilevers.16 A normal force applied to the cantilever’s tip can be easily calculated by multiplying the cantilever’s vertical deflection to its normal spring constant. Thus, the normal conversion factor can be easily determined by experimental results.

The conversion of lateral force and photodiode signal is more challenging than the normal calibration. Normally, two kinds of methods are used to calibrate the lateral conversion factor: a two-step method and a direct method. The former involves the calibration of the lateral stiffness of the cantilever and the measurement of the lateral photodiode response. This method is not straightforward and is limited in application. Unlike the calibration of the normal constant, the lateral sensitivity of the photodiode is more difficult to determine because the lateral contact stiffness between the AFM tip and the sample surface is proportional to contact radius17 and often comparable to the lateral stiffness of the cantilever and the tip,18 which significantly reduces the calibration result of the lateral sensitivity of the photodiode.19,20

In order to overcome this limitation, several methods have been put forward for lateral sensitivity measurement.21,22 A test probe with an attached colloidal sphere was successfully used to determine lateral photodiode sensitivity by loading the colloidal sphere laterally against a vertical sidewall.23 However, this kind of method is also limited in application because of difficulties in characterization of the lateral stiffness of the V-shaped cantilevers.23

In contrast with the two-step method, a one-step direct method, named wedge method, developed by Ogletree et al. is the most commonly accepted method in current use.24 This
method gets round the difficulties in the separate measurement of the lateral stiffness of the cantilever and lateral sensitivity of the photodiode. An improved wedge method developed by Varenberg et al. utilizes a commercially available calibration grating with a well-defined slope instead of the friction loops generated from different slopes, which enables calibration of all types of probes, including probes integrated with sharp or colloidal tips. A newly reported method based on direct force balances on surfaces with known slopes considers detector cross-talking and off-centered tip problems and reduces tip wear during the calibration. Direct force loading methods, including small glass fibers, a specially fabricated microelectromechanical device and magnetic forces have also been used to directly calibrate AFM lateral force measurement. Compared with the wedge method, the accuracy of calibration results with these methods is greatly affected by friction forces, and the system setup is more technically complex for the experiments.

Here, we present a new method to calibrate the lateral force measurement of the AFM using a commercially available, accurately calibrated piezoresistive force sensor. It consists of a piezoresistive cantilever and accompanying electronics, providing a standard force applied on the AFM tip for lateral force calibration. Before use, the spring constant of the piezoresistive cantilever and sensitivity of the accompanying electronics were accurately calibrated. This method may be used to directly calibrate the factor between the lateral force and the photodiode signal for cantilevers with a wide range of spring constants, regardless of their size, shape, material, or coating effects. Three rectangular cantilevers with normal spring constants from 0.092 to 1.24 N/m (lateral stiffness from 10.34 to 101.06) were calibrated. Moreover, we compared the calibration results with the theoretically calculated results based on the beam mechanics, which would yield the best results when the cantilever has a simple geometry and uniform physical properties, and the photodiode has an ideal symmetry of the normal and lateral output.

MATERIAL AND METHODS

Calibration of the piezoresistive force sensor. A piezoresistive cantilever fabricated by the standard silicon bulk micromachining technology with low scatter and drift of sensitivity can be used as a portable microforce calibration standard. The piezoresistive cantilever (Nascatec GmbH, Germany) and its accompanying electronics used in our work are commercially available. Microscopy images of the cantilever are shown in Fig. 1. Dimensions of the piezoresistive cantilever were measured as 525.8 μm in length and with an average width of 152.7 μm using microscopic image processing (under an optical microscope Olympus BX50WI with a 50× objective and Sony XC-711P CCD, providing a resolution of 0.22 μm/pixel). The top view Fig. 1(b) shows that the clamping end of the piezoresistive cantilever has a step shape with a difference of 12.5 μm on the width and a hole with a length of 15 μm on square (maybe for stress enhancement), so it is not convenient to directly calculate its normal spring constant. Therefore, in our experiment, the piezoresistive cantilever stiffness was calibrated using Cleveland’s mass loading method. We used six glass microspheres with diameters from 25.6 to 64.4 μm measured under the optical microscope, and used a glass density of 2.4 g/cm³. As shown in Fig. 1(c), the glass microspheres were released on the free end of the piezoresistive cantilever and their centers were measured for stiffness compensation due to position errors.

Experiments showed that the adhesion force between the glass microspheres and the back side of the piezoresistive cantilever was strong enough to hold the microbeads during the first mode of vibration with very low amplitude (under a humidity of 50%–60%). The first natural resonant frequency of the piezoresistive cantilever is 37.463 kHz. The stiffness of the piezoresistive cantilever was calibrated at 18.209 ± 0.471 N/m.

The next step is the force calibration of the piezoresistive sensor. The piezoresistive cantilever was mounted horizontally on a three degrees of freedom (DOF) platform, so the force applied on the cantilever was normal to its longitudinal axis. A glass substrate was attached on a Z nanopositioning stage with a resolution of 1.8 nm, which was used for the displacement increments during the calibration. On the surface of the glass substrate, a glass microsphere with a diameter of 50 μm was glued near the substrate edge used for the point of contact with the piezoresistive cantilever during the force loading. First, the nanopositioning stage was adjusted by 100 nm increments until the contact between the piezoresistive cantilever tip and the glass microsphere was achieved. The contact point on the horizontal plane was controlled by microscopy vision, while the Wheatstone bridge voltage output was used to detect the contact on the approaching direction. After the contact had been setup, a pro-
were measured using the optical microscope. The first flexure resonant frequencies were calculated as the gradient of the applied force $F$ versus the voltage output $V_p$ of the electronics, including amplifier, signal filter, and power supply.

After 20 complete loading/unloading calibration cycles, we achieved a piezoresistive force sensor sensitivity $S_p=10.361 \pm 0.267 \mu N/V$. The sensitivity $S_p=\frac{dF}{dV_p}$ is defined as the gradient of the applied force $F$ versus the voltage output $V_p$ plot.

During the force calibration of the piezoresistive force sensor, four contact points were used to test whether a position change on the width affects its sensitivity. As shown in Fig. 2, the first three contact points were located on the hemline of the trapezoidal head on the free end, on which there were two points: one on each of the left and right bottom corners, and another in the center of the line. The fourth contact point was located on the tip edge of the piezoresistive cantilever tip was approximately $5.7 \mu m$ across the full range of the piezoresistive force sensor output.

FIG. 2. (Color online) Examples of the sensitivity calibration on four different contact points (see four corresponding scaled images) on the cantilever. In which three curves with the same gradient present the contact points on the hemline of the trapezoid head on the free end of the cantilever. The fourth curve with a lower gradient denotes the calibration result when the contact point is on the tip of cantilever.

When the dimensions of the cantilever are obtained, the normal and lateral spring constants $k_n$ and $k_l$ can be calculated from

$$k_n = \frac{E_w t^3}{4 L^3},$$

$$k_l = \frac{G w t^3}{3 L (h + t/2)^2},$$

where $w$, $t$, and $h$ are the width, thickness, and tip height of the AFM cantilever, respectively.

**Testing AFM cantilevers.** Three types of AFM cantilevers with rectangular cross sections and normal force constants from 0.092 to 1.24 N/m (shown in Table I) were used: LFMR (NANO World), ContAL, and Multi75AL (Budget Sensors). Although dimensions of the cantilevers were provided by manufactures, the optical microscope (with 50x and 100x lenses) was used to determine the cantilever’s length, width, and tip height. However, the optical microscope’s resolution limitation will result in a significant error in the measurement of the cantilever’s dimension, especially its thickness. Therefore, in our experiment, the forced oscillation method was employed to determine the cantilever’s thickness based on its natural frequency. For the Euler-Bernoulli beam, if we know the resonant frequencies of the cantilevers, the thickness $t$ can be obtained from

$$t = \frac{\omega_n}{K_n} \sqrt{\frac{12p}{E}},$$

where $K_n$ is the wave number on the AFM cantilever and $p$ is its density and $\omega_n$ is the $n$th flexural resonant frequency. If $n=1$, then $K_n L = 1.8751$, where $L$ is the length of the AFM cantilever. When the dimensions of the cantilever are obtained, the normal and lateral spring constants $k_n$ and $k_l$ can be calculated from

<table>
<thead>
<tr>
<th>Tip No.</th>
<th>$L(\mu m)$</th>
<th>$h(\mu m)$</th>
<th>$w(\mu m)$</th>
<th>$t(\mu m)$</th>
<th>$f_0(kHz)$</th>
<th>$k_n(N/m)$</th>
<th>$k_l(N/m)$</th>
<th>$\alpha_0(\mu N/V)$</th>
<th>$\alpha(\mu N/V)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>228</td>
<td>15.5</td>
<td>47.8</td>
<td>0.83</td>
<td>21.84</td>
<td>0.092</td>
<td>10.34</td>
<td>1.61 ± 0.53</td>
<td>2.29 ± 0.26</td>
</tr>
<tr>
<td>2</td>
<td>449</td>
<td>17.4</td>
<td>53.4</td>
<td>2.01</td>
<td>13.64</td>
<td>0.19</td>
<td>62.27</td>
<td>11.24 ± 3.71</td>
<td>12.04 ± 1.35</td>
</tr>
<tr>
<td>3</td>
<td>229</td>
<td>17.6</td>
<td>31.4</td>
<td>2.28</td>
<td>59.49</td>
<td>1.24</td>
<td>101.06</td>
<td>18.79 ± 6.20</td>
<td>25.84 ± 2.89</td>
</tr>
</tbody>
</table>
corner of the trapezoid head on the free end. After the loading locations had been determined, the AFM tip was moved laterally to the contact location with a step of 10 nm. Under the programmed control, voltages \( V_p \) and \( V_t \), outputs of the piezoresistive force sensor and the photodiode began to be recorded at a frequency of 5 Hz when \( V_t \) reached the defined preload value of 0.01 V. During the measurement, the deflection of the AFM cantilever was controlled to keep the voltage output in the linear range of the photodiode.

Data analysis. For the top loading method, the loading force on the AFM tip can be presented as

\[
F_t = k_p \delta_p = k_t \delta_t = S_p V_p
\]

where \( k_t \) is the total lateral stiffness of the AFM cantilever-tip-contact system and \( \delta_p \) and \( \delta_t \) are deflections of the piezoresistive cantilever and AFM cantilever tip, respectively. Here, \( k_t \) can be obtained from a sum of stiffness inverses of each part: \( k_t = (1/k_{lateral} + 1/k_{tip} + 1/k_{contact})^{-1}, \) where \( k_{lateral} \) is the lateral stiffness of the AFM cantilever and its tip lateral stiffness is \( k_{tip} \) and \( k_{contact} \) is the contact stiffness between the AFM tip and the piezoresistive cantilever surface. \( k_{contact} \) is proportional to the contact radius \( r \) and often comparable to \( k_{lateral} \) and \( k_{tip}. \) \( \delta_t \) causes the lateral force calibration to be more challenging because in this case \( \delta_t \) is not equal to the real lateral deflection associated with the photodiode output. Fortunately, in our method, the lateral conversion factor is directly provided by the ratio of the applied force on the AFM tip and the photodiode’s voltage output, regardless of any knowledge of the cantilevers and the laser measuring system. For the top loading method, the force \( F_t = S_p V_p \) is applied on the AFM tip, so the conversion factor \( \alpha \) can be simply obtained from

\[
\alpha = \frac{F_t}{V_t} = \frac{S_p}{V_j}
\]

For the side loading method, in order to reduce the effects of friction force on the contact point, the loading direction is perpendicular to the piezoresistive cantilever. In this case, the lateral force conversion factor \( \alpha \) under the side loading is determined from

\[
\alpha = \frac{F_s}{V_j} = \frac{S_p L_p \cos \theta}{V_j l_p},
\]

where \( L_p \) and \( l_p \) are distances from the contact points to the clamping end of the piezoresistive cantilever for top loading and side loading, respectively (shown in Fig. 3).

However, considering that the lateral force is loaded on the side of the tip, not on the tip head of the AFM cantilever, a simple linear transformation of the factor \( \alpha \) is given by

\[
\alpha' = \alpha \left( 1 + \frac{\Delta h}{h + t/2 - \Delta h} \right)
\]

where \( \Delta h = 0.5–0.8 \mu m \) is the distance from the contact point to the tip head of the AFM cantilever and \( t \) is the cantilever thickness. For the cantilevers used in our experiments \( h = 15.5–17.6 \mu m \) and \( t=0.83–2.28 \mu m \), so an error of the factor \( \alpha \) generated from \( \Delta h \) is 2.7%–5%, which is just within an acceptable range.

![Diagram of experimental configurations](image-url)
We quantitatively compared our method with the theoretical method based on the beam mechanics. For the convenient comparison of the normal and lateral conversion factors, the photodiode sensitivity is defined as a ratio of the angular deflection of the AFM cantilever and the photodiode voltage for which the lateral inverse sensitivity is $S_l = \frac{\theta_l}{V_l}$, and the normal photodiode inverse sensitivity is $S_n = \frac{\theta_n}{V_n}$. Here, $\theta_n$ and $\theta_l$ are the normal and lateral angular deflections of the cantilever, respectively; $V_n$ and $V_l$ are the corresponding photodiode voltage outputs.

The normal spring constant $k_n$ connects the flexural deflection $x_n$ due to an applied normal force $F_n$, which can be determined by

$$F_n = k_n x_n. \quad (7)$$

So, based on the beam mechanics, Eq. (7) can be presented as

$$F_n = \frac{3}{2} k_n S_n V_n, \quad (8)$$

where $l$ is the effective length of the AFM cantilever. Therefore, the normal inverse sensitivity $S_n$ of the photodiode can be determined from

$$S_n = \frac{3}{2l} \frac{dx_n}{dV_n}. \quad (9)$$

For a cantilever with a rectangular cross section, the lateral force is related to the measured photodiode voltage $V_l$ from

$$F_l = \frac{G w t^3}{3l(h + t/2)} S_n V_l, \quad (10)$$

where $G$ is the cantilever shear modulus and $h$ is the tip height. Based on the laser beam mechanism, the displacement of the laser spot on the photodiode is decided by the reflecting cantilever’s angular deflection and the distance between the reflecting point and the spot position on the photodiode. Normally, the distance can be considered as a constant; so, based on the hypothesis that the photodiode is “rotationally symmetric,” the lateral sensitivity is assumed to be equal to the normal sensitivity. Thus, the lateral force conversion factor $\alpha_0$ can be calculated from

$$\alpha_0 = \frac{E w t^3}{3l(h + t/2)} S_n. \quad (11)$$

However, the hypothesis of photodiode symmetry is often not exactly the case due to the possible asymmetry of the laser spot shape as well as the diffraction effects from the cantilever, and may induce errors.

The beam mechanics method requires accurate knowledge of the cantilever’s elastic modulus and dimensions. As discussed below, in our experiments, the high-resolution optical microscope is used to measure the cantilever’s length, width, and the tip height, and the force oscillation method described in Eq. (1) is used to determine the thickness of the cantilever.

**RESULTS AND DISCUSSION**

As described above, two loading methods were used in our experiments: top loading and side loading. In the first case, we used Eq. (4) to calculate the lateral force conversion factor. For side loading, considering the loading position and the direction of the loading force, therefore, Eq. (5) was used to calculate the lateral force conversion factor. The inverse sensitivity $S_n$ of the photodiode described in Eqs. (7)–(9) was measured for each AFM cantilever by a linear fit on the plot of the photodiode voltage output versus displacement of the cantilevers. Here, we assumed that the photodiode inverse sensitivities $S_l$ and $S_n$ had the same value in our experiments.

The experimental results are summarized in Table I, which lists the dimensions and the mechanics of the AFM cantilevers. The dimensions, including length $L$, width $w$, and tip height $h$, were measured using the optical microscope. The first flexure resonant frequencies $f_0$ were used to determine the thickness of the cantilevers from Eq. (1). The normal spring constant $k_n$ and lateral spring constant $k_l$ were calculated from Eq. (2). The last three columns list $\alpha_0$ cal-

![FIG. 4. (Color online) [(a)-(c)] Examples of voltage outputs of the photodiode plotted vs voltage output of the piezoresistive force sensor using cantilever No. 1, No. 2, and No. 3, respectively. The blue solid circles and red lines show the data obtained from the experiments using the top loading method. The straight red lines are the linear fit of the corresponding data using the least squares method. (d) The lateral force conversion factors $\alpha$ averaged over ten experimental data repeats for each tip and the lateral force conversion factors $\alpha_0$ are calculated from Eq. (11) based on the cantilever mechanics.](Image)
culated from Eq. (11) based on the beam mechanics, and \( \alpha \) measured using the proposed top loading and side loading methods, respectively.

Each cantilever was bent laterally by the piezoresistive cantilever ten times. For each time the lateral force conversion factor was calculated as outlined in Figs. 4(a)–4(c), in which the symbol of the blue solid circles shows data obtained from the experiments using the top loading method. The straight red lines are the linear fit of the corresponding data using the least squares method; their gradients were used to calculate \( \alpha \) from Eq. (4). Then, a value of the lateral force conversion factors was averaged from the results of the ten experiment repeats. The lateral force conversion factor \( \alpha_0 \) was calculated based on the beam mechanics. Figure 4(d) shows a comparison of the lateral force conversion factors obtained from each method. The top loading and side loading results are approximately the same; the values of the side loading method are just a little higher due to the friction effect not being factored into Eq. (5). But larger differences occur between the measured factors \( \alpha \) and \( \alpha_0 \) calculated based on the beam mechanics because of cantilever dimension errors and the photodiode’s asymmetry sensitivity.

Both methods inevitably have some sources of error. For the proposed method, we need to take into account errors generated by the calibration of the piezoresistive cantilever as well as those from the lateral force calibration of AFM cantilevers, that is, variables \( S_p, V_p, \) and \( V_l \) for the top loading method, while \( \theta \) and \( l \) need to be added for the side loading method. The errors in these measurements of \( S_p, V_p, \) \( V_l, \) \( \theta, \) and \( l \) are of the order of 11%, 0.02 V, 0.02 V, 1\(^2\), and 2%. Considering the error generated from \( \Delta h, \) the maximum overall error for the calibration of the lateral conversion factor \( \alpha \) using the proposed method is 12.4%; it largely depends on the uncertainty of \( S_p. \) If an absolute force standard is used to calibrate the piezoresistive force sensor, an error of less than 6% can be expected. Using Eq. (11), the method based on the beam mechanics may yield an overall error as high as 33% with uncertainties: \( l: 2\%, w: 2\%, h: 10\%, t: 10\%, S_p: 10\%. \) For these cantilevers, the two methods actually yield similar results and errors of similar magnitude. However, as the geometry and material composition of the cantilevers become more complicated, the uncertainty of the beam mechanics method will increase significantly. Moreover, this calculation assumes that the photodiode’s lateral sensitivity and normal sensitivity are the same, which may not be true. We will test this using a colloidal tip in our future work. In contrast, the piezoresistive force sensor has several attractive features. The most significant fact is that it can provide a force standard for the direct calibration of the lateral force conversion factors without any knowledge of the photodiode or cantilever shape, dimensions, and physical properties, thereby overcoming almost all the difficulties in the calibration of the lateral force measurement.

In summary, we have presented a method to calibrate the lateral force measurement of AFM cantilevers using a piezoresistive force sensor consisting of an off-the-shelf piezoresistive cantilever and its corresponding electronics. This method was used to calibrate three rectangular cantilevers with normal spring constants from 0.092 to 1.24 N/m (lateral stiffness from 10.34 to 101.06 N/m). Compared with the calibration results of the theoretical method based on beam mechanics, the proposed approach provides lateral conversion factor values that are accurate to \( \pm 12.4\% \) or better. Moreover, this method can be used to directly calibrate the lateral force regardless of the cantilever’s geometry or knowledge of the photodiode detector, thereby enabling an accurate and direct calibration of the lateral force measurement of AFM.

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