An alternative method to determining optical lever sensitivity in atomic force microscopy without tip-sample contact

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Abstract
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Keywords
Tungsten, Atomic force microscopy, Atomic force microscopes, Point contacts, Calibration, Bending, Materials analysis, Optical microscopes, Nanomaterials, Nanotechnology

Disciplines
Mechanical Engineering | Nanoscience and Nanotechnology

Comments

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Citation: Review of Scientific Instruments 81, 073711 (2010); doi: 10.1063/1.3459886
View online: http://dx.doi.org/10.1063/1.3459886
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/81/7?ver=pdfcov
Published by the AIP Publishing
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(Received 23 April 2010; accepted 13 June 2010; published online 28 July 2010)

Force studies using atomic force microscopy generally require knowledge of the cantilever spring constants and the optical lever sensitivity. The traditional method of evaluating the optical lever sensitivity by pressing the tip against a hard surface can damage the tip, especially sharp ones. Here a method is shown to calculate the sensitivity without having to bring the tip into contact. Instead a sharpened tungsten wire is used to cause a point contact directly onto the cantilever and cause cantilever bending. Using beam theory, the sensitivity thus found can be converted to the equivalent sensitivity that would be obtained using the tip location. A comparison is presented between sensitivity values obtained from the conventional tip contact method and those derived from the wire-based technique for a range of cantilevers in air. It was found that the difference between the calculated sensitivity from the wire-based technique and the sensitivity obtained conventionally was less than 12%. These measurements indicate the presented method offers a simple alternative approach to obtain optical lever sensitivity without compromising the tip shape. © 2010 American Institute of Physics. [doi:10.1063/1.3459886]

I. INTRODUCTION

Since its invention in the early 1980s the atomic force microscope (AFM) has become one of the most powerful tools in the fields of nanoscience and nanotechnology for the preparation and analysis of materials, nanostructures, and their functionality.1−11 An important aspect in many AFM experiments is determining the optical lever sensitivity (sensitivity) which allows the bending of the cantilever to be translated into the vertical deflection (distance) units. Combining the sensitivity and the calibrated normal spring constant12−15 of the AFM cantilever allows the forces applied to the sample to be calculated. Traditionally the sensitivity is determined by pressing the tip at the end of the cantilever onto a hard surface that is assumed to not deform under the applied load, and observing the slope of the force curve.16 However, pressing the tip onto a hard surface can result in damage to the tip, especially for sharper tips and tips with functionalizations such as organic films and carbon nanotubes.12 Figure 1 shows the change in tip shape that occurred to a sharp silicon tip (series HAR5 from Nano-Science Instruments Inc.) while finding the sensitivity in the conventional manner. This change corresponded to a 70% change in the tip radius. Data from other tips with similar initial tip radii also showed shape change from the traditional sensitivity calibration. NSC15 (Mikromasch) showed a tip radius change on the order of 50% and NP-S, silicon nitride tips (Veeco Instruments, Santa Barbara) showed a tip radius change of 25%. Researchers have developed noncontact methods to determine the sensitivity. These techniques require measuring the thermal noise spectrum of the cantilever,18,19 creating calibration curves for each length of cantilever,20 changing parts of the optical system,21 or the use of colloidal tips only.22 We present here a technique that allows the sensitivity to be evaluated for any AFM tip/cantilever for which the beam bending equations can be determined, without the need for tip-sample contact or modification to a commercial microscope. In this method a sharpened tungsten wire is brought into contact directly onto the cantilever at a point away from the tip, and the sensitivity can be found from the force curve obtained with a correction made for the offset between the contact point and location of the tip. In this article, we present our investigations for commercially available rectangular cantilevers with a range of spring constants.

II. EXPERIMENTAL METHODS

The required bending of the cantilever is achieved via a point contact directly on the cantilever rather than by tip-sample contact. The point contact is achieved by a sharpened tungsten wire that is held vertically allowing the cantilever to be brought down into contact for the sensitivity measurement as shown in Fig. 2. The tungsten wire was created using conventional electropolishing, which is used widely to prepare samples in field emission and atom probe microscopy.23 To create the tungsten tip used here, a 1 cm long, 0.305 mm diameter tungsten wire was electropolished using 5% NaOH at 4 V to obtain a needle shape using a loop electrode.24

For a rectangular cantilever, beam theory can be used to calculate the change in observed sensitivity caused by moving the contact point along the cantilever and away from the location of the AFM tip. For all discussions here distances will be taken as measured along the cantilever axis from the base of the cantilever toward the end of the cantilever. There
are two different alignments of the contact point that must be taken into consideration. First, the situation where the contact point is past the laser reflection point [Fig. 2(a)], and second, the situation where the contact point is before the laser reflection point [Fig. 2(b)].

The parameter of concern is the angle of the cantilever at the laser reflection spot on the cantilever because when a force is applied to the cantilever, the cantilever bends and the reflected light moves through an angle twice the change in the slope of the cantilever at the reflection.\(^{16}\) For a rectangular cantilever, the equation for the angle of the cantilever at the laser spot \(\theta_p\), for the case where the point of contact is past the laser spot [Fig. 2(a)] is

\[
\theta_p = \frac{P}{2EI}(2xl - l^2),
\]

where \(P\) is the force applied to cantilever at the contact point, \(E\) is the elastic constant of the beam, and \(l\) is the second moment of area of the beam. Distances \(l\) and \(x\) identify the locations of the laser spot and contact point, respectively, measured from the base of the cantilever. For the case where the wire contact point is before the laser spot [Fig. 2(b)], the equivalent equation is given by

\[
\theta_b = \frac{Px^2}{2EI},
\]

assuming that the cantilever retains the same angle past the point of contact. The displacement from force applied at the contact point is given by

\[
y = \frac{Px^3}{3EI}.
\]

By rearranging and substituting Eq. (3) into Eqs. (1) and (2) yields

\[
\theta_p = \frac{3y}{(2x^3)(2xl - l^2)},
\]

\[
\theta_b = \frac{3y}{(2x^3)}.
\]

We are generally interested in the sensitivity obtained when the force is applied at the location of the AFM tip. Since the force application point (contact point of tungsten wire) is offset from the AFM tip, we need to account for the corresponding change in cantilever bending (angle) due to this offset. If \(\theta_a\) and \(\theta_w\) represent the cantilever angle at the laser spot caused by force application at the AFM tip and the tungsten wire, respectively, the percent change in the cantilever angle at the laser spot caused by this offset is given by

\[
\frac{\theta_w - \theta_a}{\theta_a} = \frac{a^3(2wl - l^2)}{w^3(2al - l^2)} - 1,
\]
It can be shown that if respective length fractions from the base of the cantilever are used as the reference sensitivity to compare the other sensitivity obtained experimentally from contact with a tungsten wire directly onto the cantilever ($S_{af}$). In order to verify the application of Eqs. (11) and (12), experiments were carried out on tipless rectangular cantilevers with a range of lengths and normal spring constants using a Dimension 3100 AFM with a Nanoscope IV controller (Veeco Instruments, Santa Barbara). The spring constants used in this study were 0.15, 2.9, and 59 N/m as measured by the Sader method. Experiments were also carried out on three silicon-probe cantilevers (HAR5 from Nanoscience Instruments Inc.) with calibrated spring constants of 35–41 N/m. Each cantilever was loaded into the AFM as normal and a frequency sweep was done to verify the resonance characteristics and check for potential problems with the cantilever. Force curves were then obtained by aligning the cantilever over the tungsten wire using the optics of the AFM and engaging on the tungsten wire. High resolution images were also captured of the alignments and used to determine the positions along the cantilever of the laser spot, AFM tip, and the tungsten wire contact for the calculations.

### III. RESULTS AND DISCUSSION

To analyze the validity of Eqs. (11) and (12) to predict the sensitivity ($S_{af}$) from the measured sensitivity ($S_{af}$), experiments with a tungsten wire were first performed on tipless cantilevers. The contact point with the tungsten wire was varied along the length of each cantilever to realize four differing length fractions ($w_f$) as listed in Table I. Table I also lists the various quantities for the cantilevers used and the obtained sensitivities $S_{af}$ (measured) and $S_{af}$ (calculated). In lieu of the sensitivity that would be obtained conventionally using an AFM tip, the sensitivity ($S_{af}$) obtained from the farthest contact point from the base of the cantilever ($w_f$) was used as the reference sensitivity to compare the other calculated sensitivity ($S_{af}$) values against. Also listed is the error associated with the sensitivity values estimated using error propagation. This includes uncertainties associated with estimating distances for the laser spot ($\pm 3\%$) and tungsten.

### TABLE I. Results from tipless cantilever sensitivity experiments. Obtained values are shown along with associated error. The error was calculated using error propagation and includes error inherent in finding the distances from images and finding the sensitivity from the force curves.

<table>
<thead>
<tr>
<th>Tipless cantilever</th>
<th>Normal spring constant (N/m)</th>
<th>Length fraction of laser spot, $l_f$</th>
<th>Farthest length fraction for contact point, $w_f$</th>
<th>Length fraction of tungsten wire position, $w_f$</th>
<th>Measured sensitivity, $S_w$ (nm/V)</th>
<th>Reference sensitivity corresponding to $w_f$, $S_w$ (nm/V)</th>
<th>Calculated sensitivity, $S_a$ (nm/V)</th>
<th>Percent difference between $S_a$ and $S_w$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.15</td>
<td>0.91</td>
<td>0.87</td>
<td>0.83</td>
<td>214.16</td>
<td>230.60</td>
<td>225.58 ± 4.28</td>
<td>2.18 ± 2.00</td>
</tr>
<tr>
<td>2</td>
<td>2.9</td>
<td>0.81</td>
<td>0.90</td>
<td>0.82</td>
<td>79.37</td>
<td>84.45</td>
<td>88.93 ± 2.65</td>
<td>−5.31 ± 3.31</td>
</tr>
<tr>
<td>3</td>
<td>59</td>
<td>0.68</td>
<td>0.82</td>
<td>0.72</td>
<td>41.34</td>
<td>47.70</td>
<td>48.19 ± 2.18</td>
<td>−1.04 ± 4.68</td>
</tr>
</tbody>
</table>

aFound using Sader’s method (Ref. 15).

bCalculated using Eq. (11) or Eq. (12) depending on if $l_f$ was larger or smaller than $w_f$.

$$\frac{\theta_a - \theta_w}{\theta_a} = \frac{a^3}{w(2a + l_f^2)} - 1,$$

where $a$ is the position of the AFM tip and $w$ is the contact position of the tungsten wire. Equation (6) is for the situation where the tungsten wire contact is past the laser spot [Fig. 2(a)] and Eq. (7) is for where the tungsten wire contact is before the laser spot [Fig. 2(b)]. Both Eqs. (6) and (7) assume that the AFM tip is at or past the laser spot, which is fairly representative of experimental conditions. It can be shown that the sensitivity for force application at the AFM tip ($S_a$) and at the tungsten wire contact ($S_w$) are related by

$$S_a = S_w \left(1 + \frac{\theta_a - \theta_w}{\theta_a}\right).$$

Using Eqs. (6) and (7) in Eq. (8) gives the following equations for the two contact situations in Figs. 2(a) and 2(b), respectively:

$$S_a = \left[\frac{a^3}{2a + l_f^2}\right] S_w,$$

$$S_a = \left[\frac{a^3}{w(2a + l_f^2)}\right] S_w.$$  

It can be shown that if $a$, $w$, and $l$ are substituted for their respective length fractions from the base of the cantilever $a_j$, $w_j$, and $l_j$, respectively, Eqs. (9) and (10) become

$$S_a = \left[\frac{a^3}{2a_j l_j - l_j^2}\right] S_w,$$

$$S_a = \left[\frac{a^3}{w_j(2a_j l_j - l_j^2)}\right] S_w.$$
wire (±2%) contact locations from the digital photos of the cantilevers as well as random error associated with estimating the sensitivity from the force curves (using the mean and standard deviation from a sample of five sensitivity estimates for each contact location). From Table I, it can be seen that the difference between the calculated sensitivities ($S_a$) for various values of $w_f$ and $S_w$ and the reference sensitivity ($S'_a$) is less than 6% for all the cantilevers tested. Figure 3(b) shows this difference visually.

Next, experiments were performed on Si probe cantilevers (HAR5) to compare the sensitivity values obtained from the tungsten wire contact method ($S_a$) with the traditional tip contact method against a sapphire sample ($S'_a$). For each HAR5 cantilever, three contact positions ($w_f$) were used to obtain measured sensitivity ($S_w$) and calculate the true sensitivity ($S'_a$). The results for three different cantilevers are shown in Table II. It was found that if the point of contact is kept past the midpoint of the cantilever length ($w_f > 0.5$), the percent difference from the calculated sensitivity to the reference sensitivity ($S'_a$) is less than 12% (Fig. 4). The data indicate that the method described here is effective in determining the sensitivity for AFM cantilevers. We note that although rectangular cantilevers were used to demonstrate the method, the technique will work for any cantilever geometry for which the bending behavior in response to a point load can be determined.

It should also be noted that one generally assumes during optical level sensitivity determinations that the contacting surface undergoes negligible deformation. In our experiments, the load needed to buckle the wire was on the order of newtons, while the loads applied in the experiments were on the order of micronewtons. The end of the tungsten wire had a typical radius of 5 µm which allowed a sufficient contact area to not damage the cantilever or the wire. Furthermore, this radius allowed for the wire end to be seen in the optics of the AFM while remaining small enough to maneuver without touching the AFM tip. It is also noted that the same tungsten wire was used for all experiments and checked between experiments for damage using an optical microscope.

### IV. CONCLUSIONS

The traditional way of finding the sensitivity of an AFM cantilever by pressing its tip against a hard surface and finding the slope of the force curve can damage sharp tips.
A method was shown to calculate the optical lever sensitivity of AFM cantilevers without having to initiate tip-surface contact. Instead a reusable sharpened tungsten wire was used to generate a point contact directly onto the cantilever and cause bending. It was found that the difference between the calculated sensitivity using this method and the sensitivity determined conventionally was less than 12% for rectangular cantilevers. The presented method offers a simple alternative approach to obtain optical lever sensitivity which is especially useful for studies where tip shape cannot be compromised.

ACKNOWLEDGMENTS

Funding for this study was provided by a grant from the National Science Foundation (Grant No. 0932573). The authors would also like to thank Curtis Mosher, Associate Scientist at the Roy J. Carver Laboratory for Ultrahigh Resolution Biological Microscopy at Iowa State University, for many helpful discussions.