Atomic force microscopy: loading position dependence of cantilever spring constants and detector sensitivity

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ABSTRACT

A simple and accurate experimental method is described for determining the effective cantilever spring constant and the detector sensitivity of AFM cantilevers on which a colloidal particle is attached. By attaching large (approx. 85 µm diameter) latex particles at various positions along the V-shaped cantilevers we demonstrate how the normal and lateral spring constants as well as the sensitivity vary with loading position. Comparison with an explicit point-load theoretical model has also been used to verify the accuracy of the method.
The quantitative interpretation of atomic force microscopy (AFM) force measurement experiments requires the conversion of raw experimental data (photodiode detector voltage versus piezo movement) to force versus piezo movement or force versus surface separation. The force, $F_z \,[\text{N}]$ is obtained as a product of the AFM photodiode-detector normal sensitivity, $S_z \,[\text{m/volt}]$, the normal spring constant of the cantilever, $k_z \,[\text{N/m}]$ and the measured voltage, $\Delta V_z \,[\text{volt}]$: \(^{1,2}\)

$$F_z = S_z \, k_z \, \Delta V_z \quad (1)$$

The detector sensitivity is determined from the hard-wall constant compliance region of the AFM force curve and there are a number of well-established methods for the determination of the cantilever normal spring constant. \(^{2-5}\)

In a recent development, the AFM colloidal probe technique was extended to measure the force acting on an emulsion droplet attached to the free end of a cantilever and driven towards either a flat substrate or a second emulsion droplet. \(^6\) As the size of an emulsion droplet attached to a cantilever can vary between 10 $\mu\text{m}$ and 80 $\mu\text{m}$, the $S_z$ and $k_z$ values in equation (1) should account for the load distribution over the cantilever-droplet contact zone. However, only the end-loaded values $S_z^e$ and $k_z^e$ (as denoted by the superscript ‘e’) can be measured experimentally, and this is done without the droplet attached. To correctly calculate the force, both of these values must be corrected to account for the off-set of the loading position when the droplet is attached. In this letter we report a simple experimental procedure in which cantilevers were loaded with latex particles to simulate droplet loading, thus allowing a consistent estimate of the appropriate spring constant and sensitivity corrections.

We conducted our investigation with a DI 3100 Dimension AFM using V-shaped cantilevers (DI NP-O tipless cantilever) typical of those employed in emulsion droplet experiments. An optical image of a latex particle probe attached to a cantilever is shown in Figure 1a and the cantilever dimensions are defined schematically in Figure 1b. The latex
particles (Duck Scientific, Copolymer Microsphere of average diameter 85 µm and density 1.05 g/cm³) were deposited initially on a Teflon coated substrate placed on the AFM sample stage from where they were picked up by the holder-mounted cantilever. Particles transferred easily from the substrate to the cantilever, most probably due to strong capillary forces. This “soft” attachment procedure allowed the particle’s position to be manipulated by pushing the particle against the inverted tip of a tapping mode cantilever chip mounted on the sample stage below the probe.

Two types of tests were performed to measure the changes in the spring constant and detector sensitivity with the particle offset position, $d$, defined as the distance between the free end of the cantilever of length $L$ and the particle center as projected in the plane of the cantilever (Figure 1b). The spring constants were estimated using the added-mass method of Cleveland. This approach requires the mass of the particle to be much greater than that of the cantilever, which was the case here. The change in the detector sensitivity was estimated from the constant compliance region of the force curves recorded when the cantilever-attached spherical particle was pressed against the solid substrate. Representative spring constant and sensitivity test data are shown in Figure 2. For consistency, the data presented were collected using the same latex particle that was attached initially at the cantilever end ($d = 0$) and then gradually pushed back. The ratio $d/L = 0.21$ corresponds to a particle whose center is at the position where the cantilever shoulders join, at $d = w/\sin\alpha$ (Figure 1b). As expected from general considerations, the cantilever spring constant increases while the detector sensitivity decreases as $d/L$ increases. We have also presented the cumulative correction factor $S_{c}^{d}k_{z}^{d}/S_{c}^{e}k_{z}^{e}$ (superscript “e” denotes the end-loaded values and “d” the offset values). The same test was repeated using several different particles and found to reproduce the data shown in Figure 2 to within ±5%.

In Figure 2 the experimental results are compared with theoretical values (solid lines) based on the V-cantilever model developed by Neumeister and Ducker. The original model
assumed loading on the front part of the cantilever \((d < w/\sin \alpha)\) only, so when the sphere is resting between the cantilever legs \((d > w/\sin \alpha)\) the model must be modified as described in the Appendix\(^8\). The upper dashed line in Figure 2 gives the spring constant dependence for the simpler case of rectangular cantilevers, for which\(^5\)

\[
\frac{k^d_z}{k^e_z} = \left(\frac{L}{L-d}\right)^3
\]  

(2)

For the V-shaped cantilever the dependence is found to be slightly below this cubic dependence (2).

The sensitivity change is proportional to the ratio of the longitudinal (pure bending) and the normal (pure deflection) spring constants \(k_z/d_k\).\(^8\) For rectangular cantilevers,\(^2\)

\[
\frac{S^d_z}{S^e_z} = \frac{L-d}{L}
\]  

(3)

Combining (2) and (3) then gives the cumulative rectangular cantilever correction factor:

\[
\frac{S^d_z k^d_z}{S^e_z k^e_z} = \left(\frac{L}{L-d}\right)^2
\]  

(4)

The corresponding V-shaped cantilever sensitivity dependence derived from the model is close to the rectangular cantilever behaviour (3). The V-shape cantilever cumulative correction (Figure 2) is also close to a quadratic dependence (4) for loading on the front part of the cantilever \((d < w/\sin \alpha)\) but slightly lower when \(d > w/\sin \alpha\).\(^8\)

The comparison of the experimentally estimated off-end load corrections and the theoretical predictions in Figure 2 shows close agreement. The slight deviation between the experimental and theoretical estimates is to be expected as the model assumes point loading, and although the particles are solid, they contact the cantilever over a finite area (Figure 1a). Our results show that as a first approximation the cumulative correction factor for undistorted droplets positioned on the front part of the cantilever, with \(d/L\) of about 0.1, will be
approximately 20%. For 50 – 80 µm diameter droplets with \( d/L \) from 0.15 to 0.25, the correction will be in the range of 40% to 50\% \(^9\). A more rigorous calculation of the loading corrections could be achieved by finite element numerical calculations,\(^3,4\) but this would require explicit knowledge of the particle-cantilever or droplet-cantilever contact area.

So far we have investigated the off-end loading corrections for normal force measurements. In addition, we found that the latex particles could also be used to measure the lateral spring constant \( k_\varphi \), by pushing the particle with a reference cantilever of known normal spring constant mounted vertically on the AFM sample stage as shown on Figure 3a. In this experiment the latex particle was fixed to the cantilever with a small amount of epoxy glue. The Dimension AFM integrated optical system and the independent movement of the sample stage allowed the precise alignment of the vertical cantilever’s free end and the center of the latex particle. After the alignment the AFM was operated in lateral force mode to oscillate the particle against the cantilever. A characteristic lateral force loop output signal for these measurements is shown in Figure 3b. The constant compliance region corresponds to the particle contacting the vertical cantilever. The latex particle’s stiffness was estimated to be always more than an order of magnitude higher than the normal stiffness of the vertical cantilever, so deformation of the particle could be neglected. Under this condition \( \Delta X \) in Figure 3b approximately equals the vertical cantilever deflection and the following simple relationship is valid:

\[
k_\varphi = \left( \frac{\Delta X}{\Delta V} \right) \left( \frac{k_{\text{cal}} R}{S_\varphi} \right)
\]

(5)

where \( k_\varphi \) [Nm/rad] is the lateral spring constant, \( k_{\text{cal}} \) [N/m] is the normal spring constant of the vertical cantilever, \( R \) is the particle radius, \( S_\varphi \) [rad/Volt] is the lateral detector sensitivity and \( \Delta X/\Delta V \) [m/Volt] is the constant compliance slope (Figure 3b). We determined \( S_\varphi \) using an independent method,\(^10\) but our approach also gives the lateral force calibration factor \( k_\varphi S_\varphi \).
In Figure 4 we compare experimental $k_\phi$ values with the predictions from the extended Neumeister and Ducker\textsuperscript{4,8} model. This time we present the absolute values of $k_\phi$, rather than its ratio with the end-loaded value, since for V-shaped cantilevers the latter approaches zero. In this calculation the sensitive parameter $Et^3$ (where $E$ is the cantilever material Young modulus and $t$ is the cantilever thickness) was inferred from the cantilever’s normal spring constant.\textsuperscript{4} The comparison in Figure 4 shows a close agreement between the experimental and point load model values and thus provides support to validate our approach.

In summary, we have developed a simple experimental procedure for estimating the normal spring constant, normal detector sensitivity and lateral spring constant variations for off-end-loaded V-shaped cantilevers as functions of load position. The results of this study are of specific relevance to work involving colloidal probe cantilevers that are widely used in direct force measurement experiments.
REFERENCES:


8) See “Appendix” EPAPS Document N for supplementary material. This document can be reached via a direct link in the online article’s HTML reference section or via the EPAPS homepage http://www.aip.org/pubservs/epaps.html

9) We note that, within experimental error, very good agreement was obtained between model calculations and experimental data in reference 6, where no load correction to the cantilever spring constant was made. This implies that the droplet on the cantilever must position itself, or distort, in such a way that the actual load correction must be significantly less than the 40-50% correction expected for a solid particle of the same size.

FIGURE CAPTIONS:

Figure 1. (Color on-line) (a) Optical image of a V-shaped cantilever loaded by a latex particle. (b) A schematic drawing of the cantilever with an offset loaded particle. For the cantilever investigated \( L = 194 \, \mu m, \ w = 20 \, \mu m \) and \( \alpha = 28.6^\circ \). The nominal normal spring constant was 0.06 N/m.

Figure 2. (Color on-line) Variations of the spring constant \( k_z/k_z^e \) (squares, blue) and sensitivity \( S/S^e \) (diamonds, green) correction factors (relative to the end-loaded values, as denoted by superscript \( e \) ) with load position \( d \), as measured for a V-shaped cantilever loaded with a latex particle. Also shown is the product of those two ratios (circles, red), which determines the conversion from diode voltage \( \Delta V \) to force \( F \) (Eq. 1). The solid lines show theoretical predictions based on the model of Neumeister and Ducker.\(^4,8\) The dashed lines show the theoretical rectangular cantilever behavior for comparison.

Figure 3. (Color on line) (a) Optical microscope image of a latex particle cantilever probe aligned with a vertically mounted cantilever of known spring constant. (b) Lateral force loop, for one cycle of oscillating the latex particle against the reference cantilever: A to C is approach toward the cantilever end with contact at point B; C to E is retraction with separation at point D.

Figure 4. (Color on-line) Experimental (squares) and theoretical\(^4,8\) (solid line) values of the lateral spring constant (Equation 5) as functions of the load position \( (d/L) \). The error bars represent the spread of the determined slopes values used for the experimental \( k_\phi \) determination (Figure 3b).
APPENDIX

To derive analytic expressions for the spring constants and sensitivity of a V-shaped cantilever, we have extended the model of Neumeister and Ducker\textsuperscript{1} (henceforth referred to as ND). In their model, the V-shaped cantilever is treated as a triangular plate (component I) attached to a pair of prismatic beams (component II), as illustrated in Fig. A1. A load or torque applied a distance $d$ from the apex of the triangle causes a deflection and/or an angular rotation of each of the two components. These can be calculated separately and then combined to obtain expressions for the cantilever spring constants.

ND only considered loading points within the triangular plate (I), so their formulae are restricted to situations where $d < w / \sin \alpha$ (cantilever dimensions are defined in Fig. 1b in the main body of the letter). However, we also model the case of a spherical probe attached between the cantilever legs, for which $d > w / \sin \alpha$. The formulae for each of these two cases are presented below, for both normal loading and lateral twisting. Those for the $d < w / \sin \alpha$ case are similar to ND, with a modification to take into account the position of the laser spot; those for the $d > w / \sin \alpha$ case are new. For an explanation of how the original formulae are derived see the Appendix of Ref. 1.

![Diagram of V-shaped cantilever decomposition](image-url)

Figure A1: Decomposition of a V-shaped cantilever into a triangular plate (I) and a pair of prismatic beams (II).
Normal stiffness and sensitivity

- **Case A**: \( d < \frac{w}{\sin \alpha} \)

We first consider the case where a normal force \( N \) is applied to the triangular plate (I). The resulting deflection \( \Delta z_I \) of the plate at the loading point is given by

\[
\Delta z_I = \frac{3N}{Et^3 \tan \alpha} \left[ \frac{w}{\sin \alpha} - 2d \right]^2 - d^2 \left[ 1 - 2\ln \left( \frac{w}{d \sin \alpha} \right) \right]
\]

where \( E \) is the elastic modulus of the cantilever and \( t \) is its thickness. [Note that there is an error in the sign of the final term in Ref. 1; as this term is negligible the error does not significantly affect results.] The change in angle \( \Delta \theta_I \) at the position of the laser spot is

\[
\Delta \theta_I = \frac{6N}{Et^3 \tan \alpha} \left( \frac{w}{\sin \alpha} - d' - d \ln \left( \frac{w}{d \sin \alpha} \right) \right)
\]

where

\[
d' = \max(d, d_{\text{spot}})
\]

and \( d_{\text{spot}} \) is the distance of the laser spot from the end of the cantilever (typically about half the length of the triangular plate). ND calculated the rotation at the loading point, but experimentally it is the rotation at the laser spot that is actually measured. The two only differ when \( d < d_{\text{spot}} \), i.e. when the loading is very close to the front of the cantilever.

The deflection \( \Delta z_{II} \) and rotation \( \Delta \theta_{II} \) of the beams (II) are given by

\[
\Delta z_{II} = NL^3 \left( \frac{2L}{Ewt^3 \cos^2 \alpha} + 3 \left( w \cot \alpha - d \cos \alpha - r \sin \alpha \right) \right)
\]

and

\[
\Delta \theta_{II} = \frac{3NL(1+\nu)}{Ewt^3 \cos \alpha} \left( \frac{w}{\sin \alpha} - d + r \cot \alpha \right)
\]

where the length \( r \) is given by

\[
r = \frac{L \tan \alpha + (w - d \sin \alpha)(1- \nu) \cos \alpha}{2 - (1- \nu) \cos^2 \alpha}
\]

Here \( \nu \) is the Poisson’s ratio of the cantilever. Note that here there is no need to take into account the position of the laser spot, as any rotation of the beams (II) will rotate all parts of the triangle (I) by the same angle.

The total deflection \( \Delta z_{\text{Tot}} \) is
\[ \Delta z_{\text{Tot}} = \Delta z_{I} + \Delta z_{II} + \Delta \theta_{II} \left( \frac{w}{\sin \alpha} - d \right) \]  

(A7)

This immediately provides the normal spring constant \( k_z \) via

\[ k_z = \frac{N}{\Delta z_{\text{Tot}}} \]  

(A8)

Similarly, the total rotation \( \Delta \theta_{\text{Tot}} \) is

\[ \Delta \theta_{\text{Tot}} = \Delta \theta_{I} + \Delta \theta_{II} \]  

(A9)

which gives the longitudinal spring constant \( k_{z\theta} \):

\[ k_{z\theta} = \frac{N}{\Delta \theta_{\text{Tot}}} \]  

(A10)

Thus the ratios of the the spring constants with the end-loaded values \( k_z^e \) and \( k_{z\theta}^e \) (as denoted by the superscript \( e \)) are given by

\[ \frac{k_z}{k_z^e} = \frac{\Delta z_{\text{Tot}}(d = 0)}{\Delta z_{\text{Tot}}} \]  

(A11)

and

\[ \frac{k_{z\theta}}{k_{z\theta}^e} = \frac{\Delta \theta_{\text{Tot}}(d = 0)}{\Delta \theta_{\text{Tot}}} \]  

(A12)

Finally, the sensitivity \( S \) is given by the ratio of the deflection \( \Delta z_{\text{Tot}} \) to the measured change in voltage \( \Delta V \), which is in turn proportional to the change in angle (i.e. \( V = a\Delta \theta_{\text{Tot}} \) for some constant \( a \)). So we have

\[ S = \frac{\Delta z_{\text{Tot}}}{\Delta V} = \frac{\Delta z_{\text{Tot}}}{a\Delta \theta_{\text{Tot}}} = \frac{k_{z\theta}}{ak_z} \]  

(A13)

The ratio of the sensitivity to the end-loaded value \( S^e \) is therefore given by

\[ \frac{S}{S^e} = \frac{k_{z\theta}}{k_z} \cdot \left( \frac{\Delta z_{\text{Tot}}}{\Delta z_{\text{Tot}}(d = 0)} \right) \left( \frac{\Delta \theta_{\text{Tot}}(d = 0)}{\Delta \theta_{\text{Tot}}} \right) \]  

(A14)

• **Case B:** \( d = \frac{w}{\sin \alpha} \)

The loading of the cantilever by a spherical probe attached between the legs of the V (Fig. 1 in the main body of the letter) is equivalent to the application of two half-loads
\( N/2 \) to the ends of two prismatic beams of reduced length \( L' \), which from trigonometric considerations is found to be given by

\[
L' = L - \left( d - \frac{w}{\sin \alpha} \right) \cos^2 \alpha
\]

(A15)

Note that the position of the centre of the sphere is somewhat behind the application points of the half loads, i.e. \( L' > (L - d) \).

In this case there is no deflection or rotation of the triangular plate alone (i.e. \( \Delta z_{\text{Tot}} = \Delta \theta_{\text{Tot}} = 0 \)), which simplifies the problem substantially. The response of the beams is derived by replacing \( L \) with \( L' \) in Eqs. A4 – A6 and setting \( w/\sin \alpha = d \) (corresponding to a load point directly at the ends of the beams), with the results

\[
\Delta z_{\text{Tot}} = \Delta z_{\text{II}} = \frac{NL^3}{Ewt^3 \cos^3 \alpha} \left( 2 - \frac{3\sin^2 \alpha}{2 - (1 - \nu)\cos^2 \alpha} \right)
\]

(A16)

and

\[
\Delta \theta_{\text{Tot}} = \Delta \theta_{\text{II}} = \frac{3NL^2(1 + \nu)}{Ewt^3 \cos \alpha} \left( \frac{1}{2 - (1 - \nu)\cos^2 \alpha} \right)
\]

(A17)

The spring constants and the sensitivity are calculated as before from Eqs. A8 and A10 – A14.

**Lateral stiffness**

- **Case A:** \( d < \frac{w}{\sin \alpha} \)

When a lateral torque \( T \) is applied to a probe attached to the triangular front part of the cantilever, the resulting twist of the triangular plate (I) is

\[
\Delta \phi_1 = \frac{3T(1 + \nu)}{Et^3 \tan \alpha} \ln \frac{w}{d \sin \alpha}
\]

(A18)

and the twist of the beams is

\[
\Delta \phi_\text{II} = \frac{3TL(1 + \nu) \cos \alpha}{Ewt^3} \left( 1 - \frac{6Lw \sin \alpha + 3w^2(1 + \nu)\cos^2 \alpha}{8L^2 + 3w^2(1 + \nu)\cos^2 \alpha} \right)
\]

(A19)

The total twist is then

\[
\Delta \phi_{\text{Tot}} = \Delta \phi_1 + \Delta \phi_\text{II}
\]

(A20)

which gives the lateral (torsional) spring constant \( k_\phi \) via
\[ k_\phi = \frac{T}{\Delta \phi_{\text{Tot}}} \] (A21)

These formulae come directly from Ref. 1.

- **Case B:** \( d \approx \frac{w}{\sin \alpha} \)

When the probe is attached between the beams that comprise the cantilever legs, there is no twisting of the triangular plate (\( \Delta \phi_1 = 0 \)); the twist is solely generated in the beams (II). This twist is derived from Eq. A19 by making the replacement \( L \rightarrow L' \) (Eq. A15), as we did for the normal stiffness calculation. This gives

\[
\Delta \phi_{\text{Tot}} = \Delta \phi_{\text{II}} = \frac{3TL'(1 + \nu)\cos \alpha}{E\omega r^3} \left( 1 - \frac{6L'w \sin \alpha + 3w^2(1 + \nu)\cos^2 \alpha}{8L_g^2 + 3w^2(1 + \nu)\cos^2 \alpha} \right) \] (A22)

\( k_\phi \) is then calculated as before from Eq. A21.

**Reference:**