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

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A simple and accurate experimental method is described for determining the effective cantilever spring constant and the detector sensitivity of atomic force microscopy cantilevers on which a colloidal particle is attached. By attaching large (approximately 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. © *2007 American Institute of Physics.* [DOI: 10.1063/1.2805518]

The quantitative interpretation of atomic force microscopy (AFM) force measurement experiments requires the conversion of raw experimental data (photodiode-detector voltage versus piezomovement) to force versus piezomovement or force versus surface separation. The force F_z (N) is obtained as a product of the AFM photodiode-detector normal sensitivity S_z (m/V), the normal spring constant of the cantilever k_z (N/m), and the measured voltage ΔV_z (V),[1,2]

$$F_z = S_z k_z \Delta V_z. \tag{1}$$

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 toward either a flat substrate or a second emulsion droplet.[6,7] As the size of an emulsion droplet attached to a cantilever can vary between 10 and 80 μm, the S_z and k_z values in Eq. (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 offset of the loading position when the droplet is attached. In this note 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 Fig. 1(a) and the cantilever dimensions are defined schematically in Fig. 1(b). The latex particles (Duke Scientific, Copolymer Microsphere of average diameter of 85 μm and density of 1.05 g/cm^3) 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.

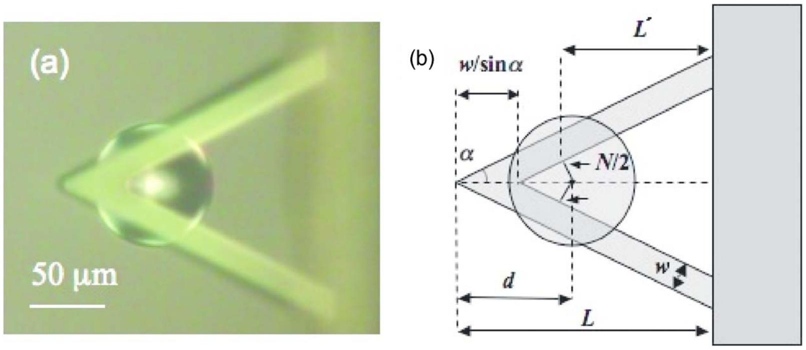

FIG. 1. (Color online) (a) Optical image of a V-shaped cantilever loaded by a latex particle. (b) A schematic drawing of the cantilever with an offset loaded particle. For the cantilever investigated $L=194$ μm, $w=20$ μm, and $\alpha=28.6°$. The nominal normal spring constant was 0.06 N/m.

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 [Fig. 1(b)]. The spring constants were estimated using the added-mass method of Cleveland.[3] This approach requires the mass of the particle to be much greater than that of the cantilever,[5] 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 Fig. 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 $d=w/\sin\alpha$ [Fig. 1(b)]. 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 $S_z{}^d k_z{}^d/S_z{}^e k_z{}^e$ (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 Fig. 2 to within ±5%.

In Fig. 2 the experimental results are compared with theoretical values (solid lines) based on the V-cantilever model developed by Neumeister and Ducker.[4,8] 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 shoulders ($d>w/\sin\alpha$), the model must be modified as described in the Appendix.[8] The upper dashed line in Fig. 2 gives the spring constant dependence for the simpler case of rectangular cantilevers, for which[5]

$$\frac{k_z^d}{k_z^e} = \left(\frac{L}{L-d}\right)^3. \tag{2}$$

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

The sensitivity change is proportional to the ratio of the longitudinal (pure bending) and the normal (pure deflection) spring constants $k_{z\theta}/k_z$.[8] For rectangular cantilevers,[2]

$$\frac{S_z^d}{S_z^e} = \frac{L-d}{L}. \tag{3}$$

Combining Eqs. (2) and (3) then gives the cumulative rectangular cantilever correction factor,

$$\frac{S_z^d k_z^d}{S_z^e k_z^e} = \left(\frac{L}{L-d}\right)^2. \tag{4}$$

The corresponding V-shaped cantilever sensitivity dependence derived from the model is close to the rectangular cantilever behavior [Eq. (3)]. The V-shape cantilever cumulative correction (Fig. 2) is also close to a quadratic dependence [Eq. (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 Fig. 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 [Fig. 1(a)]. 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%–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.

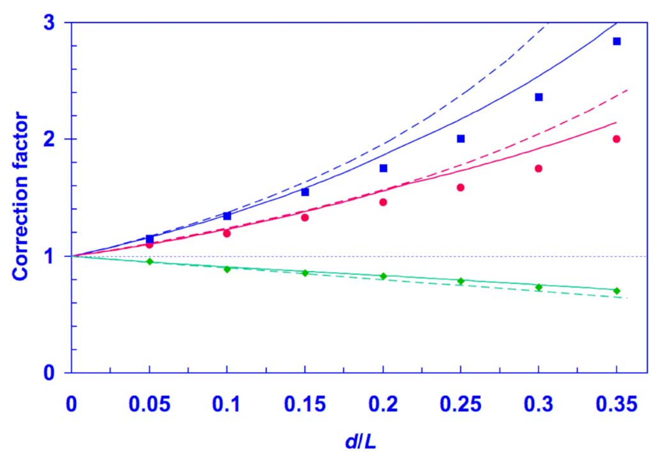

FIG. 2. (Color online) Variations of the spring constant k_z/k_z^e (squares, blue) and sensitivity S/S^e (diamonds, green) correction factors (relative to the end-loaded values, as denoted by superscript e) with load position d, as measured for a V-shaped cantilever loaded with a latex particle. Also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

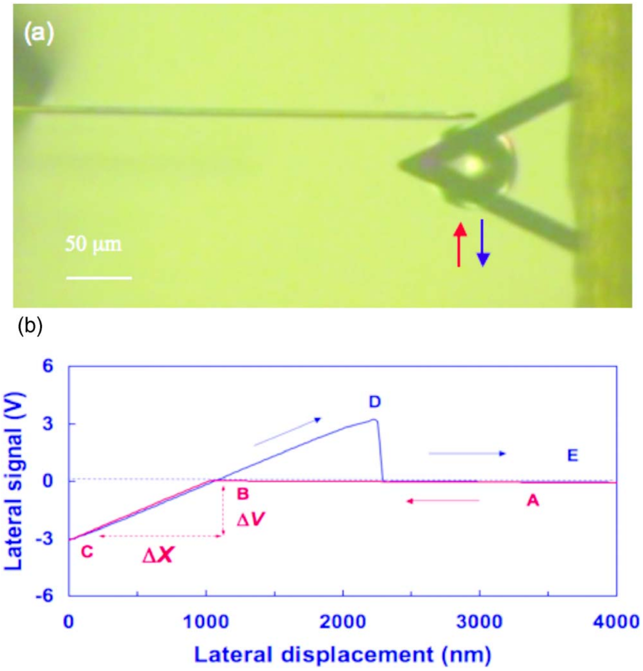

FIG. 3. (Color online) (a) Optical microscope image of a latex particle cantilever probe aligned with a vertically mounted cantilever of known spring constant. (b) Lateral force loop, for one cycle of oscillating the latex particle against the reference cantilever: A–C is approach toward the cantilever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

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_ϕ by pushing the particle with a reference cantilever of known normal spring constant mounted vertically on the AFM sample stage, as shown on Fig. 3(a). The concept of this measurement is similar to the one introduced in the apparatus developed by Ecke *et al.*[10] This time 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 Fig. 3(b). The constant compliance region corresponds to the particle contacting the vertical cantilever. The latex particle's cantilever lateral stiffness was estimated to be always more than an order of magnitude higher than the normal stiffness of the vertical cantilever. Under this condition ΔX in Fig. 3(b) approximately equals the vertical cantilever deflection and the following simple relationship is valid:

$$k_\phi = \left(\frac{\Delta X}{\Delta V}\right)\left(\frac{k^{\mathrm{cal}}R}{S_\phi}\right), \tag{5}$$

where k_ϕ (N m/rad) is the lateral spring constant, k^{cal} (N/m) is the normal spring constant of the vertical cantilever, R is the particle radius, S_ϕ (rad/V) is the lateral detector sensitivity, and $\Delta X/\Delta V$ (m/V) is the constant compliance slope [Fig. 3(b)]. We determined S_ϕ using an independent method,[11] but

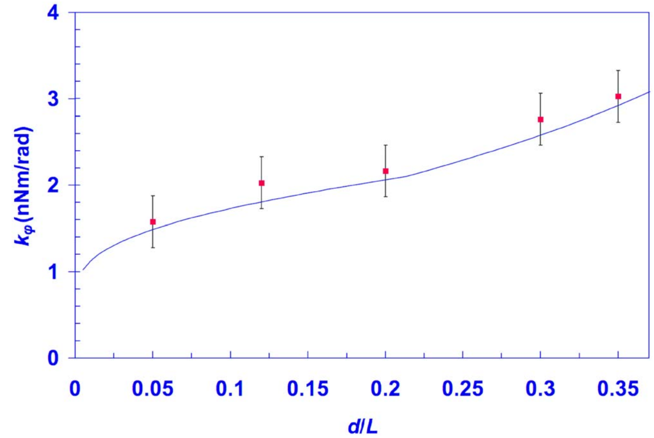

FIG. 4. (Color online) Experimental (squares) and theoretical (Refs. 4 and 8) (solid line) values of the lateral spring constant [Eq. (5)] as functions of the load position (d/L). The error bars represent the spread of the determined slope values used for the experimental k_ϕ determination [Fig. 3(b)].

our approach also gives the lateral force calibration factor $k_\phi S_\phi$.

In Fig. 4 we compare experimental k_ϕ values with the predictions from the extended Neumeister and Ducker[4,8] model. This time we present the absolute values of k_ϕ, 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.[4] The comparison in Fig. 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.

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9 We note that, within experimental error, very good agreement was obtained between model calculations and experimental data in Ref. 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.
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