Torsional spring constant measurement of a T-shaped atomic force microscope cantilever

M.G. Reitsma*, R.S. Gates and R.F. Cook

Nanomechanical Properties Group Materials Science and Engineering Laboratory National Institute of Standards and Technology 100 Bureau Dr. Gaithersburg MD 20899 USA *mark.reitsma@nist.gov

ABSTRACT

We present a microfabricated atomic force microscope (AFM) cantilever that can be used to calibrate AFM friction data. The (T-shaped) lateral force cantilever is equipped with lever-arms that facilitate the application of normal and lateral forces, allowing a calibration to be carried out *in situ* along with friction measurements. In this work, the torsional spring constant of a T-shaped cantilever has been measured using output data from two different AFM instrument platforms. Torsional spring constant values were found to be within 5% from one instrument to the other, demonstrating the robustness of the lateral force lever technique.

INTRODUCTION

Atomic force microscopy (AFM) has been the principle experimental technique used in investigations of micro-tonanoscale friction between solids for more than 20 years [1, 2]. Despite ongoing interest in this measurement technique, there is still no generally accepted method(s) to calibrate AFM instruments for quantitative friction measurement, although a few methods have been the subject of some evaluation and refinement. Two of the most commonly cited AFM friction calibration approaches are referred to here as the wedge method [3, 4] and pivot-lever methods [5]. The latter approach can be attributed to two techniques, one of which is referred to here as the pivot method [6], and the other as the lever method of Fieler et al [7], both of which are based on applying torque directly to the AFM cantilever via "Z" ramping of the AFM instrument piezo-transducer (see later). In the pivot method, a loading structure (the pivot) is typically formed by gluing an upturned cantilever (with integrated tip) onto a surface. The cantilever to be calibrated is then positioned over the pivot such that contact is established beside the integrated tip (at the same distance from the cantilever fixed end, but offset from the central long axis of the cantilever). Using the AFM instrument in force-displacement mode, Z ramping causes the cantilever to both flex and twist. However, for commercially available AFM cantilevers that typically have little or no space available beside the integrated tip of the cantilever, the application of multiple lever-arm measurements is not generally possible because of interference between the pivot and integrated tip. In the lever method, a nominally rigid beam is glued orthogonal to the cantilever long axis such that tip interference is avoided and the signal-to-noise ratio of the measurement is enhanced by the extended torsional lever-arm. A drawback of this technique is that a calibration cannot be performed in situ with friction measurements, as the beam must be removed between one procedure and the other. Recently, the pivot and lever techniques have been combined and tailored for use with a special T-shaped AFM cantilever that yields a precision AFM calibration that can be performed in situ with friction measurements [8, 9]. In the current work, we demonstrate the robustness of the lateral force (T-shaped cantilever) calibration approach by using calibration output data to determine the torsional spring constant of the lateral force cantilever itself. Results are compared on two different AFM platforms.

MATERIALS, METHODS AND RESULTS

Figure 1 shows the T-shaped "Hammerhead" devices used in this work. Figure 1(a) shows a hammerhead cantilever without a probe. Figure 1(b) shows a hammerhead device "chip" (in wafer) carrying two cantilevers, one 500 µm and the other 300 µm in length, with both cantilevers 50 µm wide and 6 µm thick. Figure 1(c) shows a ramp-tab at the back end of the chip, with fiducial marks used in calibration (see later). The devices were microfabricated using silicon-on-insulator wafers in which the device layer thickness controlled the thickness of the cantilever. The cantilevers were patterned through the device layer using photoresist and deep reactive ion etching (DRIE). The handle chip was defined by photoresist patterning and DRIE from the back side of the wafer. Final release of the cantilevers was achieved by dissolution of the buried oxide layer using hydrofluoric acid. A notched leg design (Figure 1(b)) was used to keep the handle chip securely attached to the wafer after release of the cantilevers. Finished chips were snapped out of the wafer for use.



Figure 1. Scanning electron micrographs of the prototype "Hammerhead" chip showing: (a) a hammerhead cantilever; (b) a hammerhead chip still secured in the wafer; and (c) a ramp-tab at the back end of the chip.

A typical AFM force-displacement curve is illustrated in Figure 2(a), showing a conventional rectangular cantilever and a laser spot, reflected from the cantilever back side and incident on the four-sector optical position sensitive detector (PSD) of the instrument. The corresponding measurement data represents the normal PSD output, V_N , as a function of the displacement of the surface, ΔZ , (controlled piezoelectrically), where (i) is the out-of-contact *baseline* region; (ii) is the initial contact *snap-in* region; (iii) is the *compliance* region; and (iv) is the *pull-off* region, after which the surfaces separate along (i). The 'normal sensitivity' of the AFM optical lever system is the slope of the compliance region, $S_N (= \Delta V_N / \Delta Z)$. In this case, an applied force, F_Z , can be determined from the flexural spring constant of the cantilever, k_Z , such that [10]

$$F_{\rm Z} = \frac{k_{\rm Z} \Delta V_{\rm N}}{S_{\rm N}} \tag{1}$$

Figure 2(b) shows a wing of the Hammerhead cantilever positioned over a selected fiduciary mark (which are 10 μ m apart) on a ramp chip. The lever-arm length *H* is formed through contact between the cantilever and chip edge. For the experimental setup used here, the piezo-transducer moves the surface in the *Z* direction shown ('up and down') in the same fashion as used to obtain a force-displacement curve. When the ramp chip contacts the lever-arm, the hammerhead cantilever will both twist (δ_M) and deflect flexurally (δ_Z). Since the flexural stiffness of the lateral wings of the Hammerhead are at least three orders of magnitude greater than any other stiffness property of the cantilever relevant to this work, the negligible amount of wing deflection for the type of loading shown is ignored. For the type of loading illustrated in Figure 2(b), using a cantilever of torsional spring constant, k_{ϕ} the applied force, F_Z , at a torque lever-arm length, *H*, can be written in terms of the total stiffness of the device through the total ramp displacement, ΔZ , such that

$$F_Z = \Delta Z \left(\frac{1}{k_Z} + \frac{H^2}{k_\phi} \right)^{-1}$$
(2)



Figure 2. Depiction of experimental protocols and data, where (a) shows a typical AFM force-displacement calibration with a conventional AFM cantilever; and (b) shows the lateral force (T-shaped) cantilever and the method for applying torque to the device.

Figure 3 shows representative force-displacement data for the two types of loading illustrated in Figure 2. The force-curve data (black) was acquired using the Hammerhead cantilever in the conventional way (using the cantilever probe), as depicted in Fig. 2(a). We designate the optical lever sensitivity of this data as S_N^0 . The grey torque-curve data was acquired using the type of loading illustrated in Figure 2(b). We designate the optical lever sensitivity of this data as $S_N^{(H)}$.



Recognizing that $S_N^{(H)}$ is simply a type of normal sensitivity ($\Delta V_N^{(H)} / \Delta z^{(H)}$), applying this to Equation 1 and then combining Equations 1 and 2, we have

$$\frac{S_{\rm N}^{\ 0}}{S_{\rm N}^{\ (H)}} - 1 = \frac{k_Z H^2}{k_\phi}$$
(3)

A plot of the sensitivity ratio (S_N^0/S_N^H) -1 versus k_ZH^2 should show a linear relationship with a reciprocal slope of k_{ϕ} . Figure 4 shows two separate k_{ϕ} calibrations obtained on two different AFM instrument platforms using the same (300 µm long) Hammerhead cantilever as described above. Linear regression fitting of slopes gave instrument A, $k_{\phi} = (763 \pm 46)$ nNm/rad, and instrument B, $k_{\phi} = (730 \pm 44)$ nNm/rad [11]. The better than 5% agreement between instruments is a good demonstration of the robustness of this calibration method. As a further comparison, the hammerhead cantilever was mounted into an instrumented indenter, which can make direct stiffness measurements at individual positions on the cantilever using methods described elsewhere [12]. By pressing at different points across the head of the hammerhead cantilever, a beam theory fit to experimental data gave a torsional stiffness of $k_{\phi} = [704 \pm 28]$ nNm/rad, showing acceptable agreement with AFM data (within 10% of both AFM measurements).



CONCLUSION

We have demonstrated a technique to measure of the torsional spring constant of a T-shaped cantilever using two different atomic force microscope (AFM) platforms. Results agreed to within 5%, demonstrating a clear robustness in the lateral force lever technique being developed.

REFERENCES

- 1. Mate CM, McClelland GM, Erlandsson R, Chiang S (1987) Atomic-scale friction of a tungsten tip on a graphite surface. Phys. Rev. Lett. 59(17):1942-1945.
- 2. Perry SS (2004) Scanning probe microscopy measurements of friction. MRS Bulletin. 29(7): 478-483
- 3. Ogletree DF, Carpick RW, Salmeron M (1996) Calibration of frictional forces in atomic force microscopy. Rev. Sci. Instrum. 67(9):3298-3306.
- 4. Varenberg M, Etison I, Halperin G (2003) An improved wedge calibration method for lateral force in atomic force microscopy. Rev. Sci. Instrum. 74(7):3362-3367.
- 5. Petterson T, Nordgren N, Rutland MW, Feiler, A (2007) Comparison of different methods to calibrate torsional spring constant and photodetector for atomic force microscopy friction measurements in air and liquid. Rev. Sci. Instrum. 78: 093702
- 6. Bogdanovic G, Meurk A, Rutland MW (2000) Tip friction torsional spring constant calibration. Colloids and Surfaces B: Biointerfaces 19:397-405.
- 7. Feiler A, Attard P, Larson I (2000) Calibration of the torsional spring constant and the lateral photodiode response of frictional force microscopes. Rev. Sci. Instrum. 71(7):2746-2750.
- 8. Reitsma MG (2007) Lateral force microscope calibration using a modified atomic force microscope cantilever. Rev. Sci. Instrum. 78:106102.
- 9. Reitsma MG, Gates RS, Cook RF (2008) Proceedings of the XIth International Congress and Exposition of Experimental Mechanics, June 2-5, 2008 Orlando, Florida USA
- 10. Since an AFM cantilever is typically mounted at an angle (*e.g.* approximately 10°) relative to a nominal planar surface, a geometrical correction is required to resolve force in the *Z* direction. This correction (typically 3% to 4%) is ignored for all *Z* forces in this paper.
- 11. Unless otherwise stated, all uncertainties represent 95% confidence limits (two standard deviations); except dimensional measurements using optical microscopy, where uncertainty is the optical resolution of the device used in this work.
- 12. Ying ZC, Reitsma MG, Gates RS (2007) Direct Measurement of Cantilever Spring Constants and Correction of Cantilever Irregularities using an Instrumented Indenter. Rev. Sci. Instrum. 78:063708.