Lateral force microscope calibration using a modified atomic force microscope cantilever

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A proof-of-concept study is presented for a prototype atomic force microscope (AFM) cantilever and associated calibration procedure that provide a path for quantitative friction measurement using a lateral force microscope (LFM). The calibration procedure is based on the method proposed by Feiler *et al.* [Rev. Sci. Instrum. **71**, 2746 (2000)] but allows for calibration and friction measurements to be carried out *in situ* and with greater precision. The modified AFM cantilever is equipped with lateral lever arms that facilitate the application of normal and lateral forces, comparable to those acting in a typical LFM friction experiment. The technique allows the user to select acceptable precision via a potentially unlimited number of calibration measurements across the full working range of the LFM photodetector. A microfabricated version of the cantilever would be compatible with typical commercial AFM instrumentation and allow for common AFM techniques such as topography imaging and other surface force measurements to be performed.
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Over the past two decades, the atomic force microscope (AFM) has proven to be an extensively capable tool for small-scale interfacial and materials properties studies. Investigations of small-scale friction using AFM began soon after the invention of the instrument, two decades ago.[1] Since then, the experimental technique known as friction force microscopy (FFM) and its associated field of research—nanotribology—have continued to provide important information for emerging small-scale technologies as well as insights into the origins of frictional phenomena. FFM measurements are generally performed either along the cantilever long axis (i.e., in the direction along the length of the cantilever) or perpendicular to it. While axial friction measurements—along the cantilever long axis—have been explored,[2–5] the majority of FFM measurements are made perpendicular to the cantilever long axis (and probe) in a technique called lateral force microscopy (LFM).

The central problem in quantifying friction measurements in optical lever LFM is to relate a torsional moment T on the cantilever to the lateral output from the optical position-sensitive detector (PSD) of the instrument, ΔV_L. While some promising techniques have employed force balance devices, which reportedly measure the lateral force response of a system directly,[6,7] the most commonly cited LFM calibration methods to date can be broadly classified as *wedge* and *lever methods*. Wedge methods either slide[8,9] or press[10] the AFM cantilever probe against a surface slope of well-characterized geometry, in which the mechanical response of the cantilever probe on the incline is understood in terms of force balance equilibrium. Lever methods induce torsion by displacing a lever that has been attached (usually glued) to the AFM cantilever. The lever may be a colloidal probe, for which torsion is induced via static friction between the probe and a rigid surface[11] or by pressing the equator of the sphere against another material.[12,13] Another group of lever methods displaces a beam that is attached orthogonally to both the cantilever long axis and probe.[14–17] This present work introduces a concept design for an AFM cantilever that uses the lever method of Feiler *et al.*[15] but in a way that allows for calibration and friction measurements to be carried out *in situ* and with significantly greater precision. The following demonstrates the use of the prototype AFM cantilever to generate a type of optical lever sensitivity for lateral forces, which can be used to scale raw LFM friction data for quantitative friction force measurements.

Figure 1 is a view of the experimental setup from the overhead optics of the AFM instrument used here, showing the prototype "hammerhead" cantilever. It consists of a single-crystal silicon ac-mode cantilever with a silicon cross beam glued to the free end and at right angles to the long (x) axis of the cantilever. We describe three torsion lever arms: the *experimental lever arm* is simply the probe of the cantilever (integrated tip in this case), used for friction experiments, and two *calibration lever arms*, positioned orthogonal to both the probe and cantilever long axis, used for calibration. To generate torque and minimize off-axis loading during calibration, a tungsten sphere—epoxy glued to the ramp chip—is pressed against the cross beam through the plane of the page in Fig. 1, along the "load line" shown. A simple continuum elastic analysis of the cantilever has shown that for all deflections in this work, deformation of the calibration lever arms (and glue joint between the cross beam and cantilever) is negligible.

The ramp chip shown in Fig. 1 is attached to the piezoelectric actuator (piezo) of the AFM instrument. As the piezo is ramped in the z direction, the tungsten sphere (mounted onto the ramp chip) is pressed against a calibration lever arm at a length H from the torsional axis of the hammerhead (H_R

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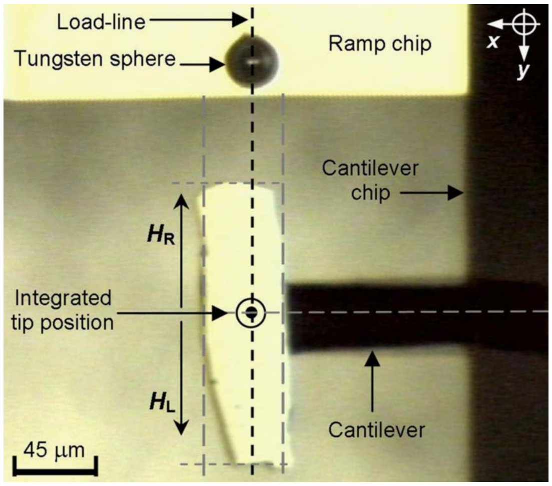

FIG. 1. (Color online) Top-down view of the experimental setup, taken through the overhead optics of the AFM instrument. Shown is the prototype "hammerhead" cantilever and ramp chip (with tungsten sphere) used to load the device during calibration. The integrated tip probe is on the underside of the cantilever.

for the right and H_L for the left lever arm). Loading in this way deflects the cantilever flexurally (in the z direction in Fig. 1) as well as torsionally (about the x axis in Fig. 1). Figure 2 shows illustrations of the hammerhead cantilever being loaded in three ways; also depicted is a laser spot (reflected from the backside of the cantilever) incident on a quadrant PSD. For the experimental data shown, the top data sets are from the normal output V_N and the bottom sets are from the lateral output V_L of the PSD. The black trace in Fig. 2 represents a single *force-displacement* curve (approach only), where for an assumed rigid probe-surface contact and elastic cantilever deflection, the *normal sensitivity* $S_N(=\Delta V_N/\Delta Z)$ is the change in normal PSD output ΔV_N as a function of surface (piezo) displacement ΔZ. In this case, an applied force F_Z can be determined from the flexural spring constant of the cantilever, k_Z, such that[18]

$$F_Z = \frac{k_Z \Delta V_N}{S_N}. \tag{1}$$

The blue traces in Fig. 2 represent *torque-displacement* curves (approach only), generated by pressing one calibration lever arm and then the other at lever-arm lengths $H_R = H_L = 72.8 \pm 0.5$ μm, measured using the overhead optics of the AFM instrument.[19] Pressing the right lever arm (positive V_L) gives the dark blue trace and the left (negative V_L) gives the light blue trace. As expected for a well-aligned system, the force-displacement curve (black trace) remains zero in the lateral output plot and both torque-displacement curves (blue traces) overlap on the normal output plot for the entire ramp range of the piezo.

Good linearity of all data in Fig. 2 means that the ratio between normal and lateral PSD output signals—the *PSD output ratio* (V_L/V_N)—remained constant over the entire range of piezo movement. Two essential requirements for the calibration technique of Feiler *et al.*[15] are that (1) all calibration data are collected within the linear range of the PSD and (2) for very small degrees of cantilever twist ($\ll 1°$), there is negligible coupling between flexural and torsional spring constants in the cantilever. Under these conditions, since a

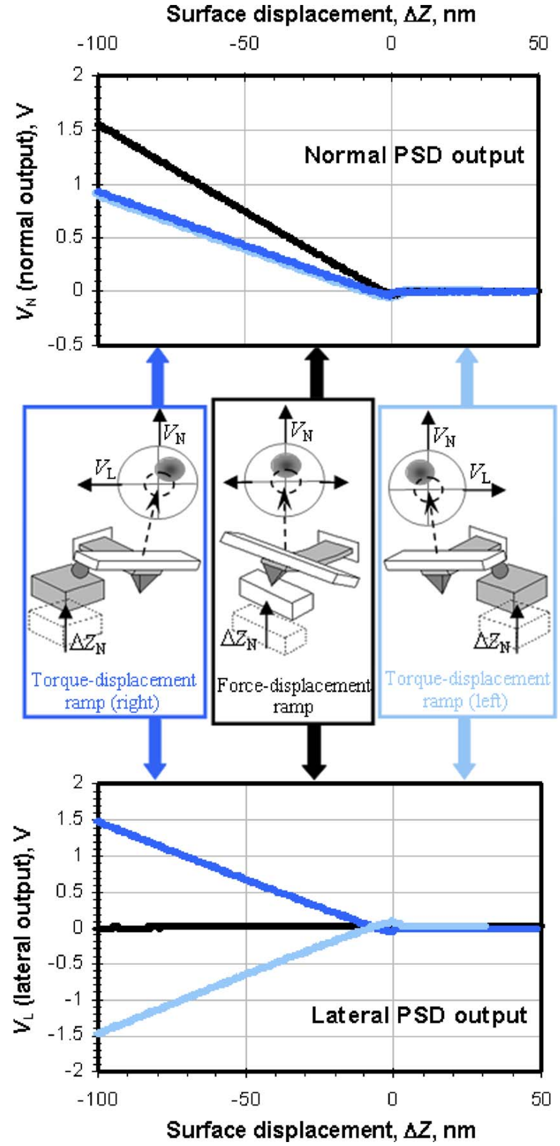

FIG. 2. (Color online) Examples of experimental calibration data (approach only) acquired using the prototype hammerhead cantilever.

force, F_Z, applied at a calibration lever-arm distance H will cause the hammerhead cantilever to deflect flexurally as well as twist, we can quantify F_Z by observing the flexural deflection response of the cantilever. In this case, we use eq. (1) to describe a lateral force, F_Z, applied to a calibration lever arm in terms of a torque, $T = F_Z H$, such that

$$T = \frac{k_Z \Delta V_N H}{S_N}. \tag{2}$$

The central problem in quantifying friction measurements in LFM is to relate ΔV_L to T. We could thus define a type of torsional parameter, which we will call the *lateral force sensitivity* (LFS) S_γ as

$$S_\gamma = \frac{\Delta V_L}{T}. \tag{3}$$

Combining Eqs. (2) and (3), we can write

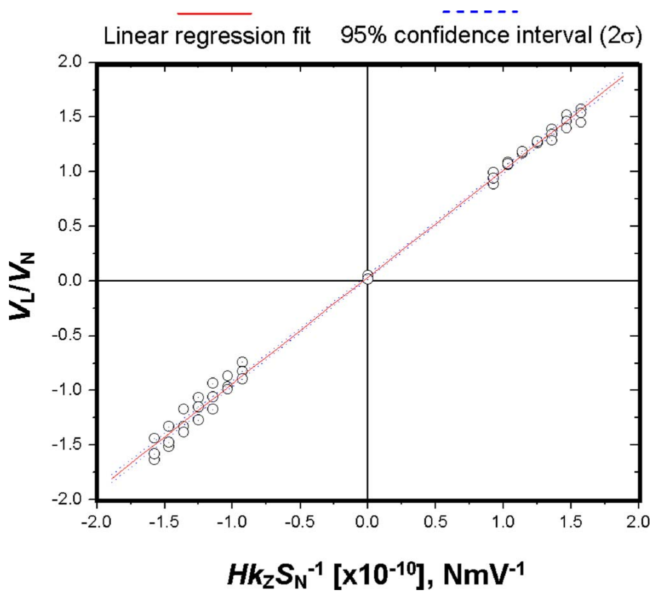

FIG. 3. (Color online) Lateral force sensitivity (LFS) calibration using prototype hammerhead cantilever.

$$\left(\frac{\Delta V_L}{\Delta V_N}\right) = S_\gamma\left(\frac{Hk_Z}{S_N}\right). \tag{4}$$

The LFS (S_γ) described above is essentially an optical lever sensitivity for lateral forces, which can be used to scale raw friction data. When measuring friction using a hammerhead-type cantilever, if the length of the cantilever probe, h, is the (z) distance from the neutral axis of the cantilever (about which twisting occurs) to the probe-surface contact where the friction force F_f is acting, then the torsional moment on the cantilever will be $T=F_fh$. The lateral output during a friction experiment, ΔV_L (i.e., "friction-loop half-width") can then be used to quantify the friction force using Eq. (3), once the LFS of the system has been calibrated. The following demonstrates a LFS calibration using the prototype hammerhead cantilever.

The flexural stiffness of the Hammerhead cantilever in Fig. 1 was measured experimentally to be $k_Z=34.8 \pm 0.2$ N m^{-1} at the position of the integrated tip using an instrumented indenter and a technique described elsewhere.[20,21] The normal sensitivity of the system, S_N, used for the LFS calibration (see abscissa term in Fig. 3), is the average S_N value for eight force-displacement curves, collected regularly throughout the experiment.

The linear plot shown in Fig. 3 was obtained by collecting a series of (Fig. 2, type) calibration measurements using different lever-arm lengths H and plotting $Hk_ZS_N^{-1}$ versus V_L/V_N. A LFS of $S_\gamma=(9.74\pm0.08)\times10^9$ V (N m)$^{-1}$ was obtained from a linear regression fit to the data.[19] Note that the PSD output ratio (V_L/V_N) of force-displacement data is plotted at $H=0$. A linear LFS plot that passes through the origin and includes both $H=0$ and $H\neq0$ data—as in Fig. 3—is a good indication of a system that is well aligned. The susceptibility of the current method to system misalignment ("crosstalk") is the subject of ongoing investigation.

Positioning uncertainty in H, measured through the overhead optics of the AFM, is presumably the major source of scatter in the Fig. 3 data. For comparison of techniques using the Fig. 3 data, the current method showed a factor of 5 improvement in precision compared to the "single-point" method of Feiler *et al.*[15]

Using the current technique, calibration and friction measurements can be carried out *in situ* by placing a ramp chip adjacent to a surface of interest in the experimental setup. A microfabricated version of the hammerhead prototype used here would offer compatibility with typical commercial AFM instrumentation, allow for AFM techniques such as topography imaging to be performed using the same cantilever, as well as the potential for true force measurement accuracy via *Système International* (SI) traceability.[22,23]

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[18] Since an AFM cantilever is mounted at a small angle relative to a nominal planar surface, a geometrical correction is required to resolve force in the z direction. For simplicity, this correction (typically 2%–3%) is ignored for all Z forces in this paper.
[19] Unless otherwise stated, all uncertainties represent 95% confidence limits, except dimensional measurements using optical microscopy, where uncertainty is the optical resolution of the device used in this work.
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