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The extended wedge method: Atomic force microscope friction calibration for improved tolerance to instrument misalignments, tip offset, and blunt probes

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One of the major challenges in understanding and controlling friction is the difficulty in bridging the length and time scales of macroscale contacts and those of the single asperity interactions they comprise. While the atomic force microscope (AFM) offers a unique ability to probe tribological surfaces in a wear-free single-asperity contact, instrument calibration challenges have limited the usefulness of this technique for quantitative nanotribological studies. A number of lateral force calibration techniques have been proposed and used, but none has gained universal acceptance due to practical considerations, configuration limitations, or sensitivities to unknowable error sources. This paper describes a simple extension of the classic wedge method of AFM lateral force calibration which: (1) allows simultaneous calibration and measurement on any substrate, thus eliminating prior tip damage and confounding effects of instrument setup adjustments; (2) is insensitive to adhesion, PSD cross-talk, transducer/piezo-tube axis misalignment, and shear-center offset; (3) is applicable to integrated tips and colloidal probes; and (4) is generally applicable to any reciprocating friction coefficient measurement. The method was applied to AFM measurements of polished carbon (99.999% graphite) and single crystal MoS2 to demonstrate the technique. Carbon and single crystal MoS2 had friction coefficients of $\mu = 0.20 \pm 0.04$ and $\mu = 0.006 \pm 0.001$, respectively, against an integrated Si probe. Against a glass colloidal sphere, MoS2 had a friction coefficient of $\mu = 0.005 \pm 0.001$. Generally, the measurement uncertainties ranged from 10%–20% and were driven by the effect of actual frictional variation on the calibration rather than calibration error itself (i.e., due to misalignment, tip-offset, or probe radius). © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4804163]

I. INTRODUCTION

Friction, though poorly understood from a fundamental standpoint, is important practically and scientifically. One of the major challenges in understanding and controlling friction is the difficulty in bridging the length and time scales of macroscale contacts and the more fundamental single asperity interactions they comprise. The atomic force microscope (AFM) is uniquely suited for fundamental studies of nanoscale single-asperity contacts, but instrument calibration challenges have limited the usefulness of the technique for quantitative tribological studies. Although numerous lateral force calibration methods have been proposed, none have been universally adopted. Munz1 discussed the benefits and drawbacks of more than 30 proposed methods in a recent review article. He describes the ideal technique as having the following attributes: (1) accuracy, (2) simplicity, (3) cross-talk compensation, (4) traceability, and (5) general applicability (any AFM, any cantilever, any tip).

The wedge method from Ogletree et al.2 is often described as the gold standard for AFM calibration.3–5 The method involves scanning along two planes of well-defined relative angle on an annealed SrTiO3 calibration substrate. With the assumption that the friction force is proportional to the normal force, they showed that standard lateral voltage loop measurements on two different “wedges” of known relative angle could be used to determine the friction coefficient and the ratio of lateral and normal calibration constants, S. This semi-direct method circumvents the need for distinct calibrations of torsional stiffness, optical lever arm, and photo-sensitive diode (PSD) sensitivity. Varenberg et al.3 replaced the nanoscale terraces of SrTiO3 with a silicon calibration grating with larger reference surfaces to allow for calibration with colloidal probes. However, as Li et al.10 point out, the validity of the moment equations breaks down if the tip is offset from the shear center of the cantilever (tip-offset). Li et al. proposed a direct method of lateral and normal force calibration by direct measurement on a calibrated diamagnetic spring system. A disadvantage of these forms of contact calibration is the need to calibrate and potentially destroy sensitive tip functionalities before making the quantitative measurements of interest. Others have developed techniques to prevent direct tip contact prior to measurement. In the test probe method from Cannara et al.,11 a colloidal probe is attached to a test cantilever of the same width and reflectivity as the target cantilever. The probe is loaded against a rigid vertical surface and the resulting piezo-displacement and PSD voltage change are used to determine the lateral deflection sensitivity, s (e.g., V/nm). The Sader method12–14 is then used to determine the cantilever spring constant, k (e.g., nN/nm) without ever making tip contact. Cannara et al.11 note that the...
uncertainty increases as the number of calibration steps increases, but showed that the test probe method agreed with the wedge method to within 5% when they accounted for in-plane bending effects. However, Li et al. argue that indirect methods are ineffective because they cannot effectively deal with tip-offset, PSD rotation, or calibration sensitivity to laser alignment.

Despite widely varying approaches to lateral force microscopy (LFM) calibration, each requires that calibration occurs before measurement. However, as Li et al. and Munz point out, standard adjustments of the PSD position, laser alignment mirror, and piezo-height between calibration and measurement can impact the optical lever arm and the overall calibration constant. This paper describes an extension of the wedge method to address error sensitivity to tip-offset, probe radius, and variations in the calibration constant during setup. This simple in situ method of lateral force calibration: (1) allows simultaneous calibration and measurement, thus eliminating prior tip damage, confounding effects of instrument setup adjustments, and the requirement of special calibration substrates; (2) is insensitive to adhesion, PSD cross-talk, transducer/piezo-tube axis misalignment, and shear-center offset; (3) is applicable to integrated tips and colloidal probes; and (4) is generally applicable to any reciprocating friction coefficient measurement.

II. MODEL

Figure 1(a) illustrates a representative friction loop obtained during reciprocated sliding on a flat tilted surface. An uncalibrated friction coefficient measurement involves determination of the lateral voltages during forward and reversed sliding. The voltage signal varies as the tip slides across the surface, so the averages and standard deviations are computed over a region in the center of the wear track that is deemed unaffected by the transient direction reversals. The friction coefficient, in arbitrary units, is calculated as $\mu = 0.5 \cdot (\bar{V}_{xf} - \bar{V}_{xr})/\bar{V}_z$. The standard deviations can be inserted into the law of propagation of uncertainty to obtain a measure of statistical uncertainty.

For calibrated friction coefficient measurements with the wedge-method, Ogletree et al. define the friction loop width, $W = (\bar{V}_{xf} - \bar{V}_{xr})/2$, and offset, $\Delta = (\bar{V}_{xf} + \bar{V}_{xr})/2$; these definitions will be retained here. The AFM is a profilometer by design and periodic calibration of the x, y, and z motions of the piezo with metrology standards is best practice. During x-movements, the instrument continuously adjusts the piezo z-height to maintain constant vertical force (PSD voltage, $V_z$). As a result, the instrument gives a direct measurement of the substrate tilt angle ($\theta$) relative to the piezo x-axis. It is important to recognize that the piezo x-axis is misaligned with respect to the cantilever x-axis by an unknown angle $\gamma$ (as shown in Fig. 1(c)) due to unavoidable manufacturing tolerances. According to the wedge method, the friction coefficient, $\mu$, and the ratio of the lateral and normal calibration constants, S, can be determined from a single tilted substrate measurement if the system is ideal. However, the effects of manufacturing tolerances are amplified at these length-scales and even slight departures from the idealized system can introduce significant calibration errors if they are unaccounted for. Ogletree et al. minimize the effects of transducer cross-talk (Fig. 1(c)), $\delta$, for example, by including a second measurement angle in the “two-slope” calibration procedure. Although simple and extremely useful, the wedge method is susceptible to other sources of calibration errors as Li et al. point out. Although there are many potential error sources in AFM systems, existing literature suggests that transducer-piezo-misalignment, tip-cantilever shear-center offset, and probe radius uncertainties are the most likely contributors to calibration error with existing methods. In an effort to reduce the potential for calibration error, we extend the wedge method to account explicitly for piezo-tilt ($\gamma$), tip-offset (O/h), and probe radius (R/h).

The free-body diagram in Fig. 1(b) illustrates the contact of a general AFM probe with a surface tilted by a wedge angle that is defined relative to the cantilever x-axis. The tip has a radius R and is offset from the shear center of the cantilever by a distance O. Balancing forces in the z-direction during
forward sliding yields

\[ F_z = F_{zf} \cdot \cos(\alpha) - \mu \cdot F_{nf} \cdot \sin(\alpha), \]  

where \( \alpha (= \theta - \gamma) \) is the angle of the surface relative to the transducer x-axis (as opposed to \( \theta \), which is the measured angle of the surface relative to the piezo x-axis). \( F_z \) is held constant during a typical AFM measurement so the normal force depends on \( F_z \) and \( \alpha \). Solving for \( F_{nf} \) gives

\[ F_{nf} = \frac{F_z}{\cos(\alpha) - \mu \cdot \sin(\alpha)}. \]  

Balancing forces in the x-direction and using Eq. (2) yields

\[ F_{xf} = \mu \cdot F_{nf} \cdot \cos(\alpha) + F_{nf} \cdot \sin(\alpha) = F_x \cdot \frac{\mu \cdot \cos(\alpha) + \sin(\alpha)}{\cos(\alpha) - \mu \cdot \sin(\alpha)} \cdot A, \]

where \( A \), a function of \( \mu \) and \( \alpha \), is defined for brevity. Balancing moments about the contact point gives

\[ M_{sf} = F_{sf} \cdot (h - R \cdot (1 - \cos(\alpha))) - F_z \cdot (O + R \cdot \sin(\alpha)), \]

\[ M_{sf} = F_x \cdot (A \cdot h - O - A \cdot R \cdot (1 - \cos(\alpha)) - R \cdot \sin(\alpha)), \]

where \( h \) is the tip height and \( O \) is the tip-offset. Repeating the force and moment balances for reverse sliding yields

\[ M_{sr} = F_{sr} \cdot (h - R \cdot (1 - \cos(\alpha))) - F_z \cdot (O + R \cdot \sin(\alpha)), \]

\[ M_{sr} = F_x \cdot (B \cdot h - O - B \cdot R \cdot (1 - \cos(\alpha)) - R \cdot \sin(\alpha)). \]

\( C_z \) and \( C_x \) are the calibration constants in the z and x directions, respectively. By definition, \( F_z = C_z \cdot V_z \) and \( F_x = C_x \cdot V_x \), where \( F_z \) and \( F_x \) are forces applied at the tip in the z and x directions, respectively. The moment, \( M_y \), to which \( V_x \) is actually sensitive, is \( M_y = V_z \cdot h \) and again by definition \( M_y = V_x \cdot h \cdot C_z \). Solving Eqs. (4) and (6) for \( V_{xf} \) and \( V_{sr} \) gives

\[ V_{xf} = \frac{V_z}{S} \bigg( A - \frac{O}{h} - A \cdot \frac{R}{h} \cdot (1 - \cos(\alpha)) - \frac{R}{h} \cdot \sin(\alpha) \bigg), \]

\[ V_{sr} = \frac{V_z}{S} \bigg( B - \frac{O}{h} - B \cdot \frac{R}{h} \cdot (1 - \cos(\alpha)) - \frac{R}{h} \cdot \sin(\alpha) \bigg), \]

recalling that \( S \) is defined as the ratio of the calibration constants \( C_z \) and \( C_x \). Ogletree et al.\(^2\) define \( W' \) and \( \Delta' \) as the derivatives of \( W \) and \( \Delta \) with respect to \( V_z \); this definition eliminates adhesion and PSD voltage offset effects. Using Eqs. (7) and (8) to solve for \( W' \) and \( \Delta' \) gives

\[ W' = \frac{1}{2 \cdot S} \bigg( A - B - \frac{R}{h} \cdot (1 - \cos(\alpha)) \cdot (A - B) \bigg) \]

\[ W' = \frac{1}{S} \frac{\mu}{\cos^2 \alpha - \mu^2 \cdot \sin^2 \alpha} \left( \frac{1}{h} \right) \cdot \left( 1 - \frac{1}{h} \cdot (1 - \cos(\alpha)) \right) \]

\[ \Delta' = \frac{1}{2 \cdot S} \left[ \left( A + B \right) - \frac{R}{h} \cdot (1 - \cos(\alpha)) \cdot (A + B) \right] - \frac{O}{h} - \frac{2 \cdot R}{h} \cdot \sin(\alpha) \bigg). \]

In the two-slope wedge method, friction measurements are made at two substrate tilt angles over a range of set point voltages. Assuming the model in Fig. 1(c) and solving Eqs. (9) and (10) for \( S \) \((C_z/C_x)\), which is constant (unless adjustments like laser position are made), we obtain the following four equations, which form the basis of the extended wedge-method:

\[ S = \frac{1}{W_1} \cdot \frac{\mu_1}{\cos^2(\theta_1 - \gamma) - \mu_1^2 \cdot \sin^2(\theta_1 - \gamma)} \times \left( 1 - \frac{R}{h} \cdot (1 - \cos(\theta_1 - \gamma)) \right), \]

\[ S = \frac{1}{\Delta_1} \left[ \frac{(\mu_1 + 1) \cdot \sin(\theta_1 - \gamma) \cdot \cos(\theta_1 - \gamma)}{\cos^2(\theta_1 - \gamma) - \mu_1^2 \cdot \sin^2(\theta_1 - \gamma)} \times \left( 1 - \frac{R}{h} \cdot (1 - \cos(\theta_1 - \gamma)) \right) - \frac{O}{h} \cdot \frac{R}{h} \cdot \sin(\theta_1 - \gamma) \right], \]

\[ S = \frac{1}{W_2} \cdot \frac{\mu_2}{\cos^2(\theta_2 - \gamma) - \mu_2^2 \cdot \sin^2(\theta_2 - \gamma)} \times \left( 1 - \frac{R}{h} \cdot (1 - \cos(\theta_2 - \gamma)) \right), \]

\[ S = \frac{1}{\Delta_2} \left[ \frac{(\mu_2 + 1) \cdot \sin(\theta_2 - \gamma) \cdot \cos(\theta_2 - \gamma)}{\cos^2(\theta_2 - \gamma) - \mu_2^2 \cdot \sin^2(\theta_2 - \gamma)} \times \left( 1 - \frac{R}{h} \cdot (1 - \cos(\theta_2 - \gamma)) \right) - \frac{O}{h} \cdot \frac{R}{h} \cdot \sin(\theta_2 - \gamma) \right], \]

where subscripts 1 and 2 represent the two wedge slopes. The slopes of the friction loop width, \( W_1' \) and \( W_2' \), in the slopes of the offsets, \( \Delta_1' \) and \( \Delta_2' \), and the wedge angles, \( \theta_1 \) and \( \theta_2 \), are determined directly from the measurements as they are in the original wedge-method. This leaves four equations and the six unknowns: \( \mu_1, \mu_2, S, \gamma, O/h \) and \( R/h \). Li et al. encountered the same problem, which motivated their direct method of calibration. We will retain all six unknowns for the time being and mathematically assess the impact of each on calibration error when neglected under typical conditions. As
Sec. III shows, we can solve the system of equations without sacrificing calibration accuracy if we assume $\gamma = 0$ and estimate $R/h$ to eliminate two of the unknowns.

III. DEVELOPMENT AND VALIDATION OF THE EXTENDED WEDGE METHOD

We must eliminate two unknowns in order to find the unique solution to Eqs. (11)–(14). Our goal here is to determine if two of the six unknowns can be eliminated without significant calibration error in realistic conditions. Typically, the researcher can reasonably estimate the quantity $R/h$ based on manufacturer reported values or direct measurement of tip radius; these values typically approach zero for integrated AFM tips and 0.5 for colloidal probes. Another unknown can be eliminated by assuming (1) $\mu_1 = \mu_2$, (2) $\gamma = 0$, or (3) $O/h = 0$; unfortunately, we cannot know how well these assumptions hold a priori since there is no way to measure or estimate these values directly. The wedge-method takes measurements at two different locations on the sample and friction coefficients on slope 1 and slope 2 can differ by 50% or more under real conditions. The $z$-piezo alignment depends on manufacturing tolerances and is expected to be of the order of $1^\circ$; $0.1^\circ$ is more typical, but alignment becomes more challenging at small length-scales. Integrated and colloidal probes are always offset from the shear center of the cantilever. According to Li et al.,\textsuperscript{10} the offset can easily approach the height even in the absence of visible offset ($O/h = 1$).

Here, we mathematically simulate an experiment with a realistic combination of frictional variation, piezo-tilt, and tip-offset. The simulated system is prescribed as: $\mu_2 = 2^{\ast} \mu_1$, $\gamma = 1^\circ$, $O/h = 1$, $R/h = 0$, and $\theta_1 = 8.5^\circ = -\theta_2$. The simulated lateral voltage responses, $V_{xt}$ and $V_{xr}$, were determined for varying values of $V_z$ and $\mu_{ave}$ using Eqs. (7) and (8). From here, the analysis is equivalent to the analysis of actual experimental data. We assume we have no prior knowledge of $\mu$, $\gamma$, or $O/h$. $W_1', W_2', \Delta_1$, and $\Delta_2$ are determined from simulated voltages per the original wedge-method (and as described in Sec. II). These simulated outputs were input into Eqs. (11)–(14) with $R/h = 0$ for an integrated probe (again, we assume we do not know $\mu$, $\gamma$, or $O/h$) resulting in five unknowns. We then reduced the number of unknowns to four using one of three simplifying cases: (1) $\mu_1 = \mu_2$, (2) $\gamma = 0$, or (3) $O/h = 0$. Next, we solved Eqs. (11)–(14) for the four remaining unknowns in each of the three case conditions.

In each case, the predicted calibration differed from the prescribed calibration. The calibration errors are illustrated in Fig. 2(a) for each case. Neglecting frictional variation (by assuming $\mu_1 = \mu_2$) resulted in gross underestimation of the friction coefficient ($\sim 85\%$). This result suggests that frictional variation has a strong effect on the calibration results and should always be accounted for; we will account for it from here on. Neglecting only tip offset ($O/h = 0$) improved the calibration, but the error remained a large fraction (63%) of the friction coefficient. Neglecting piezo-tilt ($\gamma = 0$) introduced negligible error until the actual friction coefficient approached one; interestingly, the situation does not change markedly even when $\gamma$ reaches $45^\circ$. We can conclude that only piezo-tilt can be neglected without introducing significant calibration errors when frictional variation, piezo-tilt, and tip-offset are present in realistic magnitudes.

The prior uncertainty analysis neglects optical transducer cross-talk ($\delta$) and the uncertainty in probe geometry ($R/h$). Simulating the introduction of $\delta = 1^\circ$ under the prior conditions produced no additional error. However, the introduction of a colloidal probe with 10% uncertainty in the value of $R/h$ resulted in 6.7% error. Therefore, uncertainty in $R/h$ dominates calibration error when piezo-tilt is neglected. We reconsider the previous case where $\mu_2 = 2^{\ast} \mu_1 = 0.2$, $\gamma = 1^\circ$, and $O/h = 1$ for wedges of $\theta_1 = 8.5^\circ = -\theta_2$ to map the effect of tip geometry uncertainty on friction coefficient error. A representative sharp tip ($R/h = 0.004$), an intermediate tip ($R/h = 0.04$), and a colloidal probe ($R/h = 0.4$) were analyzed. The effects of tip geometry uncertainty are illustrated in Fig. 2(b). The friction coefficient errors are negligibly small for the typical integrated tip, even in cases when tip wear or fracture changes $R/h$ by 100%. Tip characterization becomes more important for colloidal probes. A typical colloidal probe setup might include a probe radius of 5 $\mu$m $\pm$ 0.5 $\mu$m and a
cantilever thickness of 5 μm ± 1 μm. Using the law of propagation of uncertainty on the function \( R/h = R/(t/2 + 2R) \) gives \( R/h = 0.40 ± 0.016 \); the uncertainty in this case is 4% and should typically be better than 10%. In other words, if we use \( R/h = 0.4 \) in Eqs. (11)–(14) when the actual value is 0.416, the calibration error would be 2%. The extended wedge method reduces the number of unknowns in Eqs. (11)–(14) to four by using an estimated value of \( R/h \) and \( γ = 0 \). The sensitivity analysis suggests that calibration errors less than 2% can be expected under typical AFM conditions.

The sensitivity analysis shows that the tip-offset term effectively captures the effect piezo-tilt. We used a pre-calibrated microtribometer to demonstrate this effect under controlled conditions. The details of the experiment are given in the Appendix. In the standard measurement, the calibrated instrument was used to measure friction coefficient directly with an experimental uncertainty of less than 0.00219 for any one measurement. In the control measurement, the extended wedge method was used to calibrate raw voltage measurements under ideal conditions. In the test measurement, the extended wedge method was used to calibrate raw voltages under controlled conditions with \( γ = 13° \) to simulate extreme piezo-tilt. A steel sphere was slid against a grease lubricated silicon substrate and the standard measurement yielded a friction coefficient of \( μ = 0.13 ± 0.008 \). The control measurement gave \( μ_a = 0.141 ± 0.007 \) and the test measurement gave \( μ_t = 0.125 ± 0.010 \). The extended wedge method gave results well within the 95% confidence interval of the standard value for control and test measurements. Differences can be attributed to actual variations in the friction coefficient. The results validate the sensitivity study and demonstrate that the extended wedge method provides robust friction calibration when piezo-tilt is severe but assumed to be zero.

IV. DEMONSTRATION OF THE EXTENDED WEDGE METHOD WITH LATERAL FORCE MICROSCOPY

Nanoscale friction measurements were made to demonstrate the application of the method to the AFM platform. A Bruker Dimension 3100 AFM was used to make the measurements. The integrated silicon tip used here had a manufacturer (AppNano Inc.) reported stiffness of 3.8 N/m, a height of 14 μm, and a tip radius of <10 nm; tips were pre-worn against the intended substrate to reduce wear during measurement. SEM measurements of the worn tips suggest effective tip radii of 70 ± 50 nm (R/h ∼ 0.006). Repetitive, single-line scans (10 μm track at 1 Hz sliding frequency) were performed under ambient conditions (25 °C, ~35% RH) on polished (Rₐ = 150 nm) 99.999% graphite (from Kurt J. Lesker Co.), referred to as carbon, and cleaved (Rₐ < 1 nm) single crystal MoS₂, referred to as MoS₂. The substrate was wedged on a custom rotary stage with locating (via spring-loaded ball in socket joints) positions separated by 180° to produce two different wedge angles. Multiple scans were performed at normal force set-point voltages of 1, 3, and 5 V for both wedge configurations. Twenty repeat measurements (each comprising a pair of opposite wedge angles) were made on the carbon sample and ten repeat measurements were made on the MoS₂ sample; repeats were independent experiments at different sample locations.

The first step of the extended wedge method is to extract representative values of the forward and reverse voltages from each cycle (equivalent to the wedge method). To eliminate confounding transient effects near the reversals, data from the middle 5 μm of the wear track were used to calculate the mean and standard deviation of forward and reverse voltages for each friction loop. The second step is to calculate \( W \) and \( Δ \) corresponding to each combination of set point voltage and wedge angle. Mean values of \( \theta, W, \) and \( Δ \) were calculated for each friction loop as described in Sec. II. The methods described in the guide to the expression of uncertainty in measurement (GUM)¹⁵,¹⁹–²¹ were used to propagate voltage standard deviations into \( W \) and \( Δ \); averages and standard deviations are provided in Table I for reference.

The third step is to determine \( W' \) and \( Δ' \). The average of 20 values of \( W_1, W_2, Δ_1, \) and \( Δ_2 \) for carbon at each normal set-point voltage is shown in Fig. 3. Error bars represent the standard deviation in the independent repeat measurements; statistical variation within a single measurement was typically an order of magnitude smaller. \( W \) is insensitive to the wedge angle and direction while cross-talk (of unknown origins) biased the offset measurements to yield negative values of \( Δ' \) on both positive and negative wedges. For an ideal instrument, \( Δ' \) values would be equal and opposite on equal and opposite wedges.

Mean data are well fit by a linear trendline, which justifies the linear assumption in the model. Nonlinear data at low forces should be excluded from the fit when possible to minimize calibration error. Best fit slopes of \( W' \) and \( Δ' \) are provided in Table I with their statistical uncertainties. The fourth step in the calibration is to input nominal values of \( W'_1, Δ'_1, \) \( θ_1, W'_2, Δ'_2, \) and \( θ_2 \) into Eqs. (11)–(14).

The last step is to solve the system of equations. The mechanics of the method are illustrated for the carbon substrate in Fig. 4. The experimental inputs are determined directly from instrument voltage and position measurements. These experimental inputs and initial guesses for the iterated variables \( (μ_1, μ_2, \) and \( O/h) \) are inserted into Eqs. (11)–(14).
The correct values of the iteration variables are obtained when Eqs. (11)–(14) yield a common value of $S$. Assuming $\gamma = 0$ and guessing $\mu_1 = 1$, $\mu_2 = 1$, and $O/h = 0$ yields calculations of $S$ ranging from $-3$ to $28$. For convenience, we use the solver function in MS Excel to minimize the standard deviation in Eqs. (11)–(14) by iterating $\mu_1$, $\mu_2$, and $O/h$; the Excel solver is almost universally accessible and requires only a fraction of a second to find the solution. A template can be obtained by emailing a request to the corresponding author.

The values obtained for $W^\prime$, $\Delta^\prime$, and $\theta$ were used with the extended wedge-method to calibrate the system. The nominal values and standard deviations from repeat measurements are provided in Table I. Calibration with an integrated tip gave $\mu = 0.20 \pm 0.05$ for the carbon substrate and $\mu = 0.006 \pm 0.002$ with the MoS$_2$ substrate. A third cantilever with a colloidal borosilicate sphere ($\phi = 12 \mu m \pm 2 \mu m$) produced a friction coefficient of $\mu = 0.005 \pm 0.001$ against the MoS$_2$ sample. Following calibration, any friction loop width, $W$, can be converted to a friction coefficient: $\mu = S \cdot W/V_z$ (assumes no adhesion and linear behavior) whether or not the sample is wedged. This can be used to characterize friction over different phases on a single sample or determine the frictional response to environmental variations, for example. Additionally, any friction loop can be used to determine the effective friction force, $F_f$, versus frictional force. $W_f = C_1 \cdot S \cdot W$ if the normal force has been calibrated using any of the well-established techniques in the literature. This is important for load-ramped friction plots, especially in low load measurements where friction is strongly affected by adhesion and is nonlinearly dependent on normal force. To illustrate this, friction loop widths from this study have been converted to friction forces after calibrating normal force independently. Lateral forces are plotted versus normal force for carbon and MoS$_2$ in Fig. 5.

V. DISCUSSION

Li et al. suggest that indirect calibration is inherently inaccurate while semi-direct calibration accuracy is sensitive to the presence of tip-offset and non-zero probe radius. They also showed that more general semi-direct calibration models, like the one described here, contain more than the four unknowns allowed by the two-slope wedge measurement. The extended wedge-method described here retains the simplicity of the two-slope wedge measurement while accounting for tip-offset and non-zero probe radius.

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**TABLE I.** Input data and results for calibration of carbon and MoS$_2$ friction measurements. Confidence intervals for input data reflect the standard deviation. Confidence intervals for output results ($\mu$, $S$, O/h) reflect the experimental uncertainty based on Monte Carlo simulation of input statistics. Interestingly, the frictional uncertainties, which range from 15% to 20%, are comparable to the frictional uncertainties obtained with traceable microtribology measurements.

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**Carbon, integrated tip #2**

| $\theta_1$ (°) | $\theta_2$ (°) | $W_1$ | $W_2$ | $\Delta'_1$ | $\Delta'_2$ | $\mu$ | $S$ | O/h |
| $-8.930$ | $7.299$ | $0.0011$ | $0.0008$ | $-0.007$ | $0.039$ | $0.006$ | $6.221$ | $-0.113$ |
| $\pm0.406$ | $\pm0.008$ | $\pm0.0002$ | $\pm0.0004$ | $\pm0.001$ | $\pm0.004$ | $\pm0.001$ | $\pm0.154$ | $\pm0.004$ |

**Carbon, integrated tip #3**

| $\theta_1$ (°) | $\theta_2$ (°) | $W_1$ | $W_2$ | $\Delta'_1$ | $\Delta'_2$ | $\mu$ | $S$ | O/h |
| $-5.827$ | $7.049$ | $0.0006$ | $0.0010$ | $-0.044$ | $-0.025$ | $0.005$ | $6.024$ | $0.214$ |
| $\pm0.095$ | $\pm0.131$ | $\pm0.0001$ | $\pm0.0001$ | $\pm0.001$ | $\pm0.002$ | $\pm0.001$ | $\pm0.569$ | $\pm0.020$ |
found that tip-offset, $O/h$, piezo-tilt, $\gamma$, and PSD rotation, $\delta$, have unique effects on cross-talk. However, we also found that the tip-offset term captures the effects of piezo-tilt and PSD rotation with negligible error under most circumstances; conversely, piezo-tilt and PSD rotation do not generally capture the effects of tip-offset. Traceable and controlled microtribometry demonstrated that $13^{\circ}$ of piezo-tilt had no significant effect on extended wedge method calibration when the tip-offset term was used to account for the piezo-tilt. Experimental uncertainties were 8% of the measurement and were driven by statistical variations in the friction coefficient; i.e., frictional uncertainty was almost entirely due to actual frictional variation.

AFM measurements were conducted to demonstrate the application of the method to typical lateral force studies. Calibration of an integrated Si probe against carbon gave a friction coefficient of $\mu = 0.2$, a sensitivity $S = 5.4$, and a tip-offset $O/h = 0.5$. The nanoscale friction coefficient of carbon is consistent with the typical macroscale response of carbon in ambient conditions.

$MoS_2$ gave friction coefficients of $\mu = 0.005$, a sensitivity $S = 6.2$, and $O/h = -0.1$. The mean friction coefficient of single crystal $MoS_2$ is quantitatively consistent with the results from Zhao and Perry who measured $\mu = 0.007$ for single crystal $MoS_2$ against a silicon nitride AFM probe at 35% RH. The nanoscale friction coefficient of single crystal $MoS_2$ appears to be 1/40th the typical macroscale friction coefficient of sputtered $MoS_2$ in similar conditions. Follow-up measurements with highly oriented pyrolytic carbon yielded similarly low friction, so the low friction observed for single crystal $MoS_2$ during AFM measurements was likely driven by the idealized structure of the material. These low friction coefficients may have resulted from the absence of wear, the absence of reactive edge sites, or the absence of transferred fragments to the tip.

The calibration sensitivity, $S$, was statistically larger ($p < 0.0005$) during $MoS_2$ testing than during carbon testing. Differences in cantilever properties may have caused the difference, but we have found that laser realignment can itself change $S$ by 20% or more; similar observations were noted previously by Munz and Li et al.

The strong effect of laser position on calibration may have contributed here and is the primary reason we began exploring strategies of in situ calibration. Additionally, $O/h$ changed by 0.6 (equivalently $30^{\circ}$) when we changed from tip #1 to tip #2. Since this term serves as a "catch-all" for tip-offset, piezo-tilt, and optical cross-talk, we cannot know which factors contributed. Based on the simulation described in Sec. III, tip-offset, piezo-tilt, and optical cross-talk, if each acted alone to cause the 0.6 change in $O/h$, would have caused 0.0%, 0.9%, and 1.1% calibration errors, respectively. This demonstrates the degree to which $O/h$ captures the effect of the other unknown cross-talk contributors in realistic conditions. The calibration errors of the extended wedge-method are, therefore, expected to be far less than the statistical variations in most cases.

Colloidal probes cannot be used with the wedge-method due to the nanoscale size of the substrate terraces and the inability of the solution method to accommodate a non-zero tip radius. The extended wedge-method can be used with any substrate of interest and accounts explicitly for $R/h$, but requires that $R/h$ be estimated and used during calibration. The uncertainty in $R/h$ dominates the calibration errors of the method. We showed through simulation that the errors are negligible for integrated tips, even when $R/h$ estimates are off by 500%. However, the friction coefficient error becomes much more sensitive to errors in $R/h$ for colloidal probes. Fortunately, $R/h$ is easily measured or estimated for colloidal probes and is generally known to within 5%, which results in less than 2% error in the friction coefficient calibration (Fig. 3). The AFM measurements of $MoS_2$ were repeated with a colloidal borosilicate glass sphere ($\phi = 12 \mu m \pm 2 \mu m$) to demonstrate applicability of the method. The friction coefficient of $MoS_2$ against colloidal glass was $\mu = 0.005$, which is consistent with the results against silicon and previous results from Zhao and Perry against silicon nitride.

This suggests that the friction of single crystal $MoS_2$ is relatively insensitive to the countersurface material and contact scale.

Although the calibration errors are expected to be small, the calibration results are sensitive to experimental and statistical uncertainties. There are three experimental error contributors in the extended wedge method: $\theta$, $\Delta'$, and $W'$ (one on each wedge); sample-sample variations dominated other contributors to uncertainty in every case studied here. In the case of the integrated carbon measurements $\theta$, $\Delta'$, and $W'$ caused 25%, 42%, and 33% of the combined uncertainty, respectively. Since $\Delta'$ depends on $\theta$ (local topography) and $W'$ (friction), we can conclude that frictional variation contributed just more than angular variation. In the case of the integrated $MoS_2$ measurements, $\theta$, $\Delta'$ and $W'$ contributed 17%, 21%, and 62% of the combined uncertainty, respectively; frictional variation dominated uncertainty in this case. In the case of the colloidal $MoS_2$ measurements, $\theta$ contributed only 5% to combined uncertainty, which was again driven by frictional variation. The reduction in the angular uncertainty in this case is likely driven by the averaging effect of the large contact area. In general, frictional variation was the largest contributor to experimental uncertainty which ranged from 10% to 20% across the study. Interestingly, the AFM uncertainty
results were consistent with those from traceable microtribometry measurements where efforts were made to minimize frictional variability.

VI. CONCLUSION

In conclusion, this paper describes an extended wedge-method of AFM friction force calibration, which uses a new solution strategy to calibrate standard two-slope wedge measurements. The method:

1. can be applied to any substrate, which enables in situ calibration to eliminate prior tip damage and changes in the optical path between calibration and measurement,
2. requires no on-the-fly compensation and only requires that the X and Z axes have been calibrated according to standard metrology practices,
3. is insensitive to adhesion, PSD offset, PSD cross-talk, transducer/piezo-tube axis misalignment, and shear-center (tip) offset,
4. is applicable to integrated and colloidal probes, and
5. exhibited uncertainties of 8% during traceable microtribometry and 10%–20% in the AFM measurements due primarily to actual frictional variation for both cases.

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APPENDIX: VALIDATION AND AFM DATA

1. Controlled microtribometry

The AFM is virtually impossible to calibrate with traceable force standards and the small size of its components increases the difficulty of alignment and location of critical transduction elements. Consequently, the AFM is a poor platform for conducting validation efforts. Calibrated instruments on the other hand provide a known reference standard. Experiments were initially conducted on the custom-designed reciprocating microtribometer shown schematically in Fig. 6 to demonstrate the method and its insensitivity to severe piezo-tilt. The microtribometer is conceptually analogous to an AFM; it has normal and lateral force sensing elements along with normal and lateral actuation and feedback. Both nanopositioning stages (PI piezoelectric stages) are factory calibrated to uncertainties of 1 nm (z) and 5 nm (x). Similarly, the six-channel load cell (ATI Industrial Automation) has been factory calibrated to an uncertainty of 3 mN. There is one very important difference between the AFM and microtribometer; while the lateral signal of the AFM is proportional to frictional torque, the lateral signal of the microtribometer is proportional to the frictional force. O/h and R/h are always zero when the output is independent of frictional torque. This instrument has a friction coefficient uncertainty of less than 0.002 under all conditions used here, and is therefore an ideal platform to validate the extended wedge-method under controlled conditions.

A 1/4 in. 440 C sphere was mounted to the load-cell as the probe. A flat of polished silicon was adhered to a tilt stage which was mounted to the reciprocating stage. Silicon was used to minimize the effect of topography on the measurement and lithium grease was used as a boundary lubricant to minimize wear and promote frictional stability. The sliding speed was fixed at 500 μm/s over a 200 μm reciprocation track. Prior to testing, a tilt stage was adjusted to set the wedge angle, \( \theta \). The normal force was varied between 1 and 5 V (corresponding to 1–5 N) via the z-piezo stage. Capacitance measurements of the x and z piezo-stage positions were used to determine \( \theta \). Friction voltage loops were obtained for each set-point voltage at both wedge angles (\( \theta_1 \) and \( \theta_2 \)), as would be done for the traditional wedge method.

In the standard measurement, the factory calibrated instrument was used to collect friction coefficient with the collection of each voltage loop on the microtribometer. In the

![FIG. 6. Schematic illustration of the custom-microtribometer for the case where the piezo-actuator and load transducer are (a) aligned and (b) misaligned by \( \gamma = -13^\circ \) to simulate the effect of extreme piezo-tilt.](image-url)
control measurement, the extended wedge method was used to calibrate raw voltage measurements, corresponding to a microtribometer configuration shown in Fig. 6(a). To simulate severe piezo-tilt, in the test measurement, the load cell was misaligned relative to the piezo-stage axes by an angle $\gamma = -13^\circ$, as shown in Fig. 6(b); per the extended wedge-method, we assume $\gamma = 0$.

The mean and standard deviation for the raw standard measurement were measured as $\mu = 0.131 \pm 0.008$. The solution of the extended wedge method for the control measurement yielded $\mu = 0.141$, $S = 1.037$, and $O/h = 0.0042$; $O/h$ is physically zero in the aligned case, but appears to be non-zero due to unavoidable manufacturing tolerance stacking ($\gamma \sim 0.2^\circ$). A mean friction coefficient of $\mu = 0.125$ was obtained for the test measurement. Each agrees well with calibrated values (S was known a priori to have a value of 1 from factory calibration of load cell).

The errors in the friction coefficients from the extended wedge method itself are less than 0.05% (per the method described in Sec. III); i.e., if measured outputs had no random, time-dependent, or load dependent fluctuations, and were measured exactly. The uncertainty in this measurement is, therefore, only a function of the uncertainties in $W$, $\Delta$, and $\theta$ as described earlier. Since the extended wedge solution is numerical, experimental uncertainty can only be determined numerically. Each value of $W$, $\Delta$, and $\theta$ was perturbed to high and low limits ($\bar{x} \pm \sigma_x$) and a solution was obtained for each combination ($2^9 = 64$ possibilities). The mean of 64 friction coefficients is the nominal value and the standard deviation is the uncertainty. The experimental uncertainties for the aligned and misaligned cases were 0.007 and 0.01, respectively. These values are consistent with the scatter in the actual friction coefficient data, suggesting that actual frictional variations dominated the calibration uncertainty (as opposed to calibration error). The 95% confidence interval for the aligned case is [0.127, 0.155] and [0.105, 0.145] for the misaligned case; in both cases, the actual friction coefficient lies within the 95% confidence interval of the calibration. The results suggest that the frictional variation dominated the effect of piezo-tilt on calibration error; severe piezo-tilt was observed to have no impact on calibrated friction values obtained from the extended wedge method.

2. AFM measurement data

Lateral force microscopy data for experiments with integrated probes are provided in Table II.

![Table II](image-url)

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References: