Editor's Note: Starting with this issue, TLT is pleased again to be publishing the winning abstracts written by student members of STLE that participated in the Student Poster Competition held at STLE's 2012 Annual Meeting & Exhibition last May in St. Louis, Missouri (USA). This year's contest set an all-time record with 46 posters on display from students representing major universities in the U.S. and abroad, including South Korea, China, Czech Republic and France.

The quality of the submissions was top-notch and represented the hard work of the students and their professors. In addition, the society wishes to thank the poster judges: Drs. Mitjan Kalin (University of Ljubljana), Thomas Scharf (University of North Texas), Jun Qu (Oak Ridge National Laboratory), John Lucero (NASA), Daniel Nelias (INSA) and Greg Sawyer (University of Florida) for an outstanding job in evaluating all the posters, as well as Dr. Robert Erck of Argonne National Laboratory and STLE student member Sukbae Joo (Texas A&M) for organizing the event.

Please feel free to contact the authors—perhaps you can offer some guidance, a mutual research or work opportunity or even employment within your organization. This is your chance to review tomorrow's ideas and talent before they become today's products and competitors. Also, be sure to check out our short video interviews with the poster winners, which will be included in the digital version of TLT (available at **www.stle.org**).

Methods in Characterization of Nanoscale Friction in Solid Lubricants

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INTRODUCTION

The limited applicability of conventional lubricants in extreme environments has motivated the use of solid lubricant materials. Solid lubricants accommodate low friction and low wear sliding by forming low shear-strength surface films (called tribofilms). Although conventional wisdom suggests a liquid-like bulk shear process, recent studies suggest that the films and shear are confined to within 10 nm of the surface.[1]

These films are too thin and have proven difficult to detect and even more difficult to probe mechanically. Scanning probe microscopy (SPM) is perhaps the only technique with sufficient surface sensitivity. However, quantitative SPM-based indentation and lateral force measurements are notoriously difficult due to inherent calibration challenges. The goal of this work is to leverage existing calibration strategies to improve quantitative reliability of SPM-based friction measurements (lateral force microscopy, LFM).

EXPERIMENTAL

Two-Slope Lateral Force Calibration

The wedge method of lateral force calibration[2] utilizes calibration substrates with known wedge angles to extract values of friction coefficient and the lateral force calibration constant. Once friction loop width and offset (W and Δ) are experimentally determined (*see Figure 1*), friction coefficient is evaluated using the expression

$$\mu + {}^{1}/_{\mu} = {}^{2\,\Delta'}/_{W' \sin 2\alpha}$$

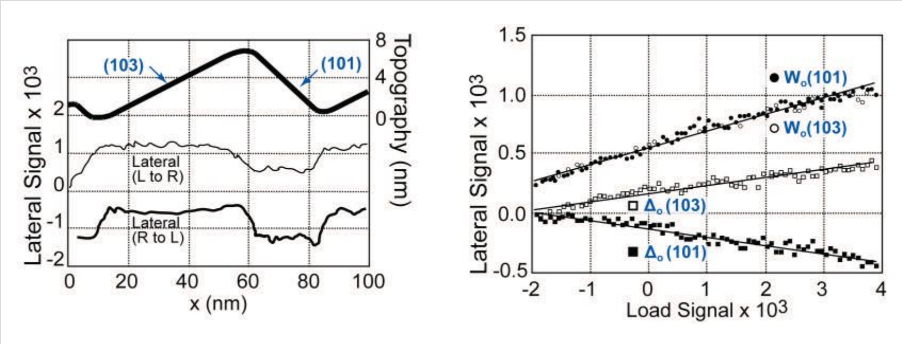


Figure 1 | **The wedge method relies on a two-sloped calibration standard ($SrTiO_3$) to obtain corresponding friction loops (*shown left*). Loop width (W) and offset (Δ), when measured for different loads on two facets (101 and 103) of $SrTiO_3$ (*shown right*), are used to determine friction coefficients and calibration constant. *Adapted from* [2]**

The two-slope modification of the wedge method is critical for eliminating photodiode cross-talk, which is always present but unknowable. A calibration standard with known geometries is used to calibrate the lateral force. The calibration is then applied to subsequent lateral force measurements as it would for any other instrument.

In addition to photodiode induced crosstalk, piezo-stack misalignment and tip shear offset introduce significant but unknown errors into SPM calibration (*see Figure 2*). Perhaps more important, however, the traditional approach reasonably assumes that the calibration constants are constant. Nominally identical repeat measurements on three independent AFMs have revealed exceptionally poor measurement repeatability. This is due to the fact that the calibration constants are sensitive to several factors related to standard instrument and sample setup including z-piezo voltage, cantilever positioning, and the optical path of the laser. We have developed a method for simultaneous calibration and measurement to eliminate the confounding effects of standard SPM measurement variables.

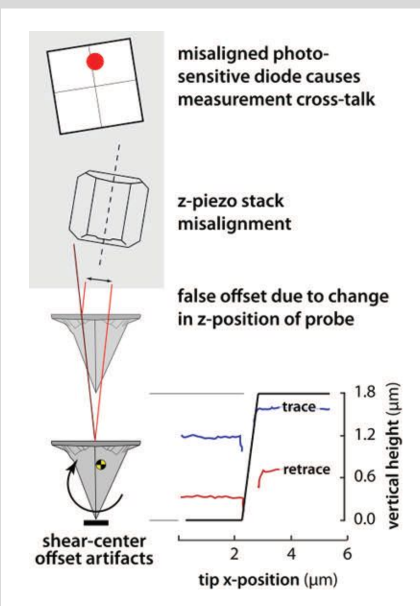


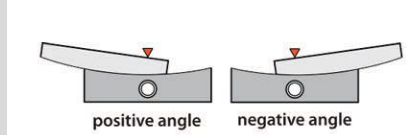
Figure 2 | **Piezo-stack misalignments and complex optical paths with an SPM introduce stochastic uncertainties in measurement of friction.**

In situ Calibration and Measurement

Using force equilibrium at the tip-substrate contact, (similar to the formulation in [2]) friction loop width and offset as a function of applied normal load (W' and Δ') are written in terms of the wedge angle, friction coefficient and ratio of normal to lateral calibration constants C_x/C_y (*see Figure 3*). In addition to the wedge angle α, a misalignment angle γ is included to accommodate the 'effective' misalignment of the transducer (tip-shear offset, piezo-stack misalignment, etc.).

Experimentally, independent friction measurements (μ_1 and μ_2) are made *on the sample* at two different wedge angles (α_1 and α_2), as illustrated in Figure 3. The combination of two equations (described by Figure 3) for two wedge angles yield four equations with four unknowns, namely C_x/C_y, γ, μ_1 and μ_2, where, C_y is calibrated directly. It is worth noting that the proposed method does not impose any restriction on the values of measured friction coefficients at the two wedge angles (i.e., μ_1 and μ_2 are not constrained).

Performing *in situ* calibration on the surface-of-interest during the measurement eliminates the need for constant calibration constants. Further, incorporation of the effective transducer misalignment angle γ into the model significantly reduces the uncertainty associated with the instrument-related complexities illustrated in Figure 2.



$$\left(\frac{C_x}{C_y}\right) \cdot W' = \frac{\mu}{\cos^2(\alpha-\gamma) - \mu^2 \cdot \sin^2(\alpha-\gamma)}$$

$$\left(\frac{C_x}{C_y}\right) \cdot \Delta' = \frac{\sin\alpha \,.\, \cos(\alpha-\gamma)\,(1+\mu^2)}{\cos^2(\alpha-\gamma) - \mu^2 \cdot \sin^2(\alpha-\gamma)}$$

Figure 3 | ***In situ* calibration involves friction measurements on the sample at two wedge angles. Force-equilibrium equations are solved to obtain friction coefficient and calibration constants.**

In order to validate the proposed method, the process of calibration and measurement was carried out on a well-characterized, custom-built microtribometer (*see Figure 4*), where calibration values for the load cell are known as *a priori*. Measurements were made at two wedge angles across a range of normal loads and measured values of W' and Δ' were used to evaluate calibration constants. Evaluated values of C_x/C_y extracted from the experimental data were determined to be within 2% of the manufacturer reported value, with friction coefficients μ_1 and μ_2 as 0.106 and 0.099; and misalignment angle γ -0.16°.

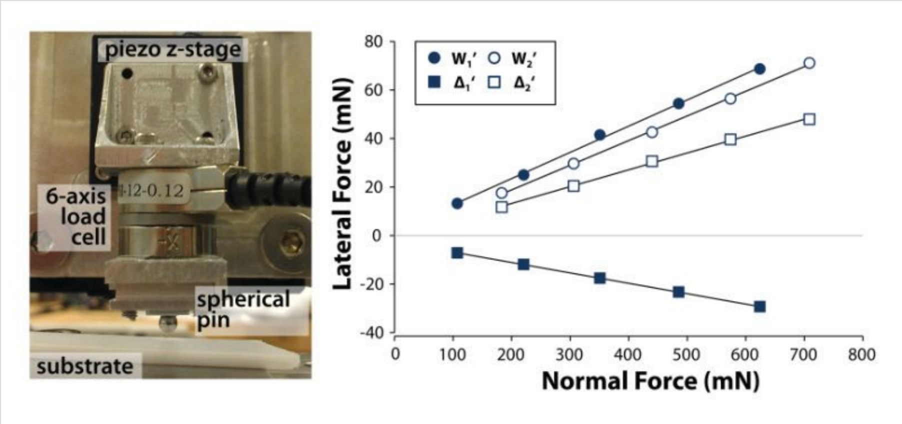


Figure 4 | **Validation of the method was done with a custom tribometer, fitted with a 6-channel load cell of known force sensitivity (*shown left*). Friction loop width and offset was measured for two substrate wedge-angles at different loads (*shown right*).**

NANOTRIBOLOGICAL CHARACTERIZATION

The *in situ* lateral force calibration method is applied to quantify changes in tribological properties of MoS_2 coatings as a result of sliding-induced tribofilm formation. Figure 5 (*see inset*) represents a typical uncalibrated friction versus load graph used for evaluating W,' illustrating qualitative differences in friction between worn, unworn and single crystal MoS_2. Quantitative measurements performed using the in situ technique on worn and unworn coatings show reduction in friction coefficient due to sliding.

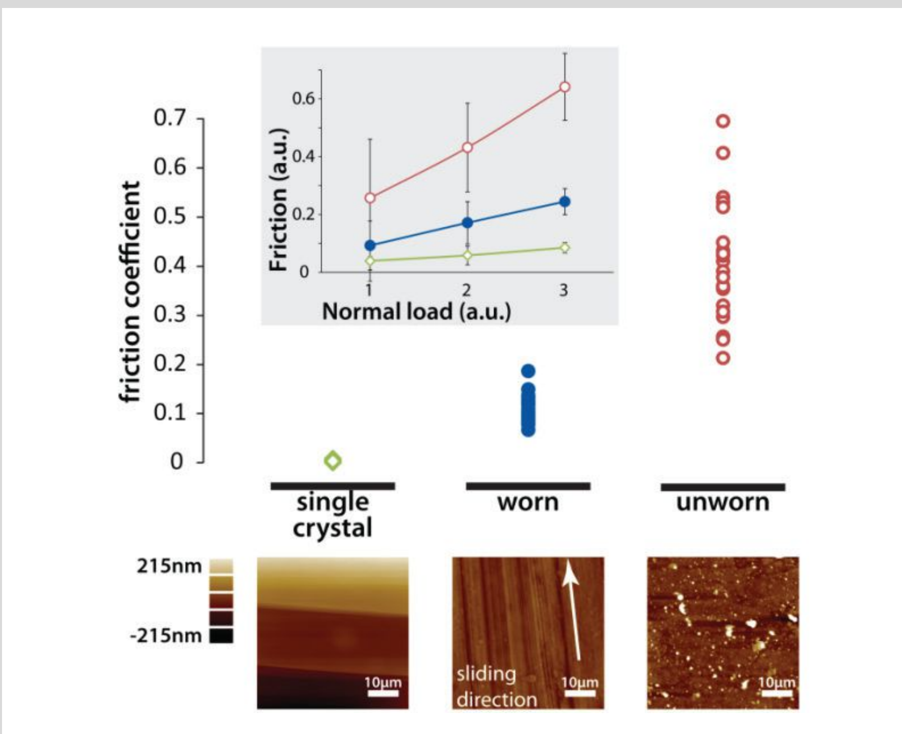


Figure 5 | **Friction coefficient measured with the *in situ* method for worn and unworn sputtered MoS_2 and single crystal MoS_2 reflect changes in substrate microstructure due to the formation of sliding-induced tribofilms (which mimic the basal structure of single crystal MoS_2). Corresponding AFM topography images are also shown. Force-units in the inset figure are arbitrary (a.u.).**

Thin-ordered tribofilms are seen to mimic the perfectly-ordered microstructure of single crystal MoS_2, which exhibits the lowest value of friction (*see Figure 5*). These results illustrate the applicability of the *in situ* calibration method as applied to the measurement of nanoscale friction at solid lubricant tribofilms.

CONCLUSIONS

While SPM-assisted friction measurements are key to characterizing solid lubricant tribofilms, inherent instrument uncertainties make existing calibration and measurement quantitatively unreliable.

To overcome SPM measurement challenges, an *in situ* method of nanoscale lateral force measurement is presented, which performs a single-step calibration and measurement of nanoscale friction.

The method is validated using a well-characterized microtribometer. Applicability to the measurement of nanofriction in solid lubricants is illustrated by quantitative LFM friction coefficient measurements and the clear effects of surface structure and tribofilm presence on nanoscale friction.

ACKNOWLEDGMENTS

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