RHK Technology Brief
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The Atomic Force Microscope as a Critical Tool for Research in Nanotribology

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Introduction

The atomic force microscope (AFM) is an invaluable tool for studying single-asperity contacts and their structural and tribological properties, including friction, adhesion, wear, and topography[1]. We use an RHK Technology variable-temperature (VT), ultra-high vacuum AFM (UHV350 AFM/STM) with SPM100 and AFM100 electronics to characterize micro- and nano-scale interfaces in controlled or UHV environments (base pressure < 10^{-10} mbar). The unique "beetle"-type[2] design of this AFM exhibits low thermal drift, making it possible to image pristine surfaces (vacuum-cleaved or annealed/prepared in situ) with a high level of mechanical stability. We regularly obtain low-noise, atomic lattice resolution images (in contact mode) for a variety of materials. **Figure 2** shows room temperature deflection and stick-slip images of the NaCl (100) surface, cleaved in situ at < 10^{-10} mbar. The slope-shaded topographical image shows steps and terraces, as well as a screw dislocation (top right region). A smaller, 10nm scan on a terrace reveals the NaCl lattice.

A large portion of our most recent work relies on the VT capability of the UHV350. At elevated temperatures, we have observed the NaCl surface undergo remarkable topographical changes, including the creation and motion of dislocations. **Figure 3** shows images that illustrate this behavior, which is enhanced in the lateral force image. In addition, we have achieved stick-slip friction at low temperatures. While some thermal drift occurs in an image (**see Figure 4 on next page**), the stick-slip behavior persists, and it is possible to correct for the small amount of distortion due to drift. With little known about the processes that govern stick-slip at the atomic scale, the UHV350 enables us to study this phenomenon with tremendous flexibility and control over experimental conditions.

In addition to stick-slip behavior, measuring and interpreting friction as a function of applied load is critical for developing a complete understanding of the physics and tribological properties at sliding interfaces. In the sections below, we describe our basic procedure to obtain and interpret friction versus load data using the RHK system.

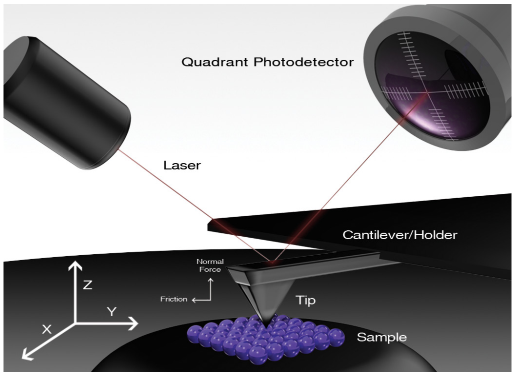

Figure 1: *Schematic diagram of an atomic force microscope with optical beam deflection detection of cantilever deformation due to normal and lateral forces on the tip.*

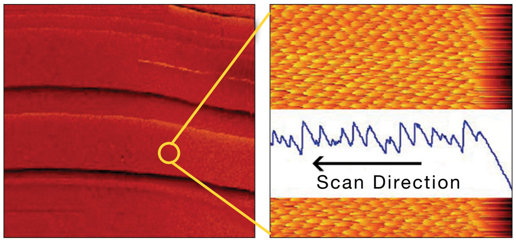

Figure 2: *2x2 μm² deflection (left) and 10x10 nm² friction trace (right) showing stick-slip behavior with periodicity equal to the lattice constant of NaCl (5.65 Å); raw data is shown without filtering.*

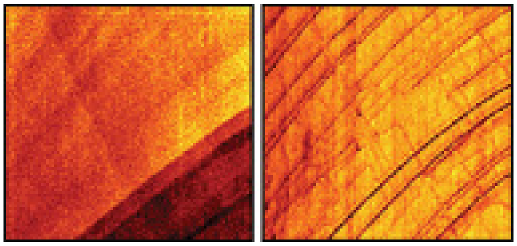

Figure 3: *2x2 μm² topography (left) and lateral force (right) images, revealing screw dislocations at 450 K.*

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Selecting an Appropriate Cantilever and Probe

For friction measurements, we typically purchase commercially-available, contact mode (~10-20 kHz flexural resonance frequency) cantilevers with integrated tips composed of either bare silicon or silicon nitride, or coated with a second material chosen based on specific properties, such as hardness or conductivity. In some experiments (e.g., to measure the lateral deflection sensitivity from lateral force-distance curves), we glue colloidal probes to the tip end of a commercial cantilever. In addition, we coat tips with amorphous carbon coatings of different thicknesses using the 100-200 kV electron beam in a transmission electron microscope (TEM).

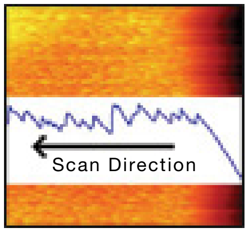

Figure 4: *Lateral force image and line trace at 128 K.*

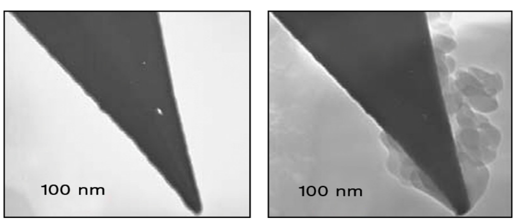

Figure 5: *TEM micrographs of a tungsten carbide-coated Si AFM tip before (left) and after (right) scanning on a Si surface show how contamination and tip erosion can occur.*

TEM is ideal for measuring the dimensions and tip shape of each cantilever, as well. normal and torsional spring constants are easily characterized with the flexural and torsional Sader methods[3-4],eliminating a large source of error that results from using nominal manufacturer values. Optical microscopy is sufficient for measuring the length and width of a cantilever and the distance of the tip to the free end of the lever (at x10 and x40 magnification). Using TEM, we determine the cantilever thickness, as well as the height and position of the tip (making certain that it is centered along the width of the lever), its radius at the apex, and its structure (i.e., whether it is amorphous or crystalline). **Figure 5**. TEM micrographs of a tungsten carbide-coated Si AFM tip before (left) and after (right) scanning on a Si surface show how contamination and tip erosion can occur.

In general, a tip is chosen based on its shape and material properties, and we perform TEM before and after an experiment to record how much a tip may have changed. It is very common for tip erosion to occur during sliding. **Figure 5** shows an example of an AFM tip that has been contaminated and worn by scanning. It is crucial to use TEM or some other tip characterization method (e.g., an image-tip reconstruction algorithm) to track variations in tip shape.

Aligning the Laser and Centering the PSD

Compliant cantilevers are preferred for their increased sensitivity to lateral forces relative to stiffer levers. Accordingly, it is important to align the laser as close to the free end of the cantilever as possible. It is also critical to center the laser spot on the four-quadrant photosensitive detector (PSD) both to minimize uncertainty in an experiment and to avoid truncating the laser spot at the edge of the photodetector. **Figures 6a and 6b (on next page)** show the optical arrangement and the dependence of (lateral) deflection sensitivity, or change in signal per change in displacement, on the position of the laser spot with respect to the center of the PSD. In general, both the normal and lateral deflection sensitivity decreases with increasing PSD offset (e.g., as it moves from position 1 to 3 in **Figure 6a**). This occurs as a result of the non-uniform intensity distribution of lasers, regardless of truncation.[5] **Figure 7 (on page 5)** helps illustrate this effect schematically with a cantilever undergoing a (small) sinusoidal oscillation about its long axis. The output response depends on the laser spot position on the PSD. Thus, while the lateral signal output for the laser spot at position 1 represents the true

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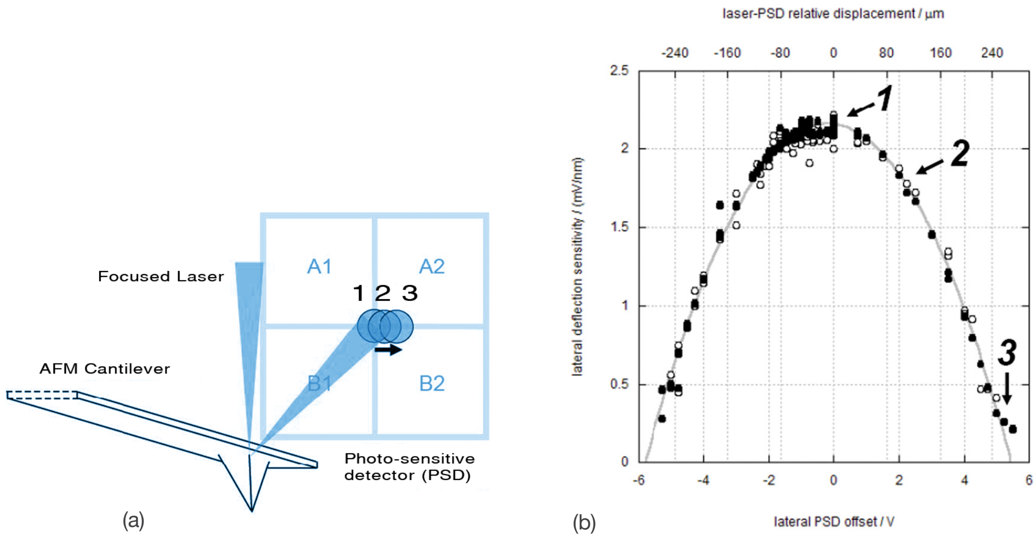

Figure 6: ***(a)*** *Arrangement of cantilever, focused laser beam and photo-sensitive detector (PSD) in an atomic force microscope (AFM) (beam waist drawn to scale relative to PSD); the initial laser spot position may be offset from the center of the PSD, indicated by positions 1, 2 and 3.* ***(b)*** *Lateral deflection sensitivity versus PSD offset signal and displacement with positions 1, 2 and 3 indicated; data were obtained from the slope of the contact region in a lateral force versus distance curve for a colloidal probe against a rigid, vertical surface.*[5]

mechanical behavior of the cantilever, a lateral offset of the laser spot relative to the center of the PSD (positions 2 and 3) yields a distorted response.

Unlike other commercial UHV AFMs, the PSD in the UHV350 may be adjusted both vertically and horizontally with internal stepper motors. This feature permits us to make fine alignments to the position of the reflected beam relative to the center of the PSD, thereby maximizing detection sensitivity and minimizing measurement uncertainty in an experiment.

Force-Distance Curves and Ramping Applied Load

With the point spectroscopy mode in the software, we take normal force-versus-distance curves to measure the pull-off force between the tip and surface and to establish the range of loads to investigate in a friction versus load study. There are several ways to ramp the load in an experiment: (1) by varying the setpoint with the feedback on, (2) by changing the z-displacement, or (3) by modulating the setpoint with the feedback gains near zero. The best approach depends on the scan size and surface properties (e.g., rms roughness, chemical inhomogeneity, etc.) Greater inhomogeneity or topographical variation calls for feedback control. For stick-slip measurements, we minimize the gains or disable the feedback loop entirely, because cantilever torsion is accompanied by small but physical changes in the normal signal which can induce a feedback response that interferes with the measurement. The image spectroscopy mode controls the setpoint or z-voltage ramp during images.

The geometry of every AFM requires that the cantilever be tilted by some angle relative to the sample. In the RHK AFM, this angle is 22.5°. Cantilever tilt produces different effects depending on the type of measurement, such as in-plane contributions to damping in intermittent contact mode AFM.[6] Similarly, for contact mode measurements, including friction versus load and force-distance measurements, this tilt causes the relative position of the tip and surface to change depending on the applied load. Consequently, when a surface is chemically or topographically inhomogeneous, or if different load measurements must be recorded on the same scan line, it is necessary to use cantilever tilt compensation.[7] While other systems require external control to achieve compensation, it is accomplished quite easily

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with the RHK AFM by selecting an asymmetric image size in the software (where the y-scan size can be greater than the x-size) or with the electronics (separate vs. ganged x and y range switch on the SPM100).

Analyzing Friction Versus Load Data

Friction is half of the difference between the lateral signal in the trace and retrace directions. In the RHK software, we use the "difference" option in image processing to subtract the trace and retrace images and then plot the line-by-line average (e.g., software "y-average" option) of the difference versus the line-by-line average of the deflection signal. This ability to analyze data in real time helps expedite the process of measurement and to make informed decisions about the direction to take in an experiment. Later, we employ a combination of several calibration schemes to convert normal and lateral signals to nano-scale forces with optimal accuracy and precision. The data can then be compared and contrasted with other data or fit to a contact mechanics model. The shape of a friction versus load curve depends on multiple factors: the geometry of the contact (e.g., sphere on flat, flat on flat, etc.); the contact mechanics (elasticity and deformation); and the shear strength, which results from chemical interactions (making and breaking bonds) and can exhibit a separate load dependence. Several well-established models exist which describe single-asperity contact mechanics (or, more specifically, how contact area depends on load). The most well-known of these models are the Derjaguin-Muller-Toporov (DMT) model[8],the Johnson-Kendall-Roberts (JKR) model[9], and an approach that combines DMT and JKR into a single transition model.[10-11]

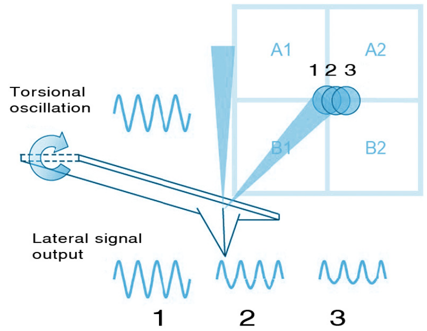

Figure 7: *Schematic representation of the cantilever undergoing a torsional, sinusoidal oscillation at different lateral offsets of the laser spot relative to the PSD. The lateral signal (A1+B1-A2-B2) output from the PSD depends on laser spot position. If it is offset from the center of the PSD, the output signal will be distorted from the sinusoidal signal that represents the true mechanical behavior.*

Calibrating Forces

To calibrate normal and lateral spring constants, we use the Sader methods mentioned above. The normal force calibration factor is the normal spring constant divided by the magnitude of the slope of the repulsive region in a normal force distance curve taken on a hard surface. There are several lateral calibration methods discussed in the literature; however, Ogletree's variable-load wedge method[12] is the most widely accepted approach. If this wedge method is not possible or if the probe cannot be scanned on a calibration surface, the lateral force calibration may be obtained in a fashion similar to normal force calibration. Lateral force-distance measurements with a colloidal probe against the vertical edge of a GaAs sample (**Figure 8**) can be used to calculate the lateral deflection sensitivity in an experiment.[5]

Regardless of the normal or lateral force calibration method, the laser must be centered on the photodetector to optimize precision. An offset of the spot from the center of the PSD leads to increased measurement uncertainty, as

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shown in **Figure 9**. Lateral calibration is a crucial step in friction force microscopy in which the best results can be compared quantitatively with other work. Choosing both the appropriate calibration method and an AFM with the most control over tip-sample geometry and detector alignment produces the most robust measurements.

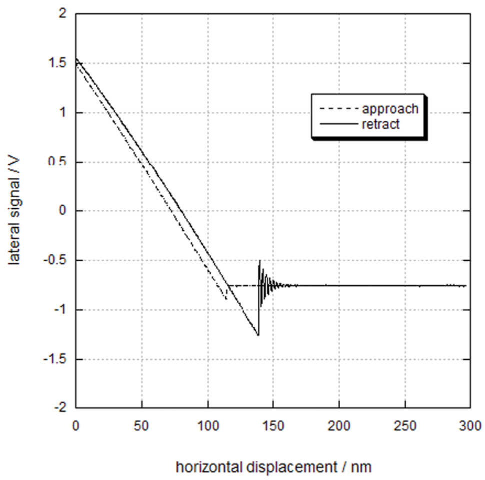

Figure 8: *Lateral force-distance curve for a colloidal probe against a vertically-oriented (110)plane of a GaAs crystal.*

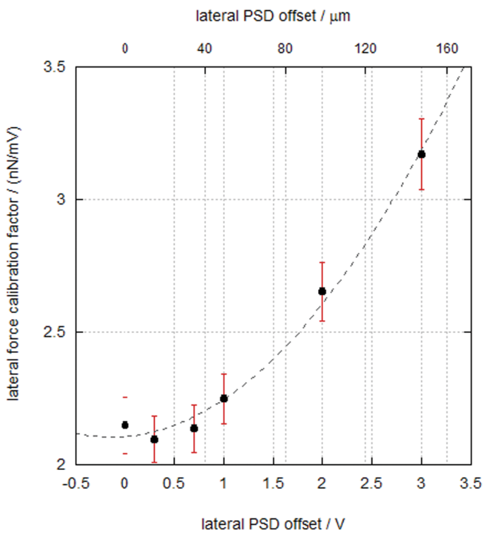

Figure 9: *The lateral force calibration factor versus lateral PSD offset in volts (out of ~6V) and microns (where the width of each PSD sector is 1.3 mm). Dashed line is a parabolic curve fit. The absolute uncertainty is a percentage of the calibration value and, therefore, increases with increasing displacement of the laser spot relative to the center of the PSD.*

Conclusion

Until recently, tribology has remained a mere phenomenology, having lacked the tools that are necessary for studying single-asperity contacts. In the last decade or so, the AFM has enabled scientists to peer into the atomic regime and begin to uncover the fundamental mechanisms of frictional energy dissipation. To explore the contribution of each mechanism, we need controlled environments, thermal and mechanical stability, and the ability to take accurate and precise measurements. The RHK design makes all of this possible.

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