Features of static and dynamic friction profiles in one and two dimensions on polymer and atomically flat surfaces using atomic force microscopy

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Abstract. In this paper we correlate the Atomic Force Microscope probe movement with surface location while scanning in the imaging and Force versus distance modes. Static and dynamic stick-slip processes are described on a scale of nanometres to microns on a range of samples.

We demonstrate the limits and range of the tip apex being fixed laterally in the force versus distance mode and static friction slope dependence on probe parameters. Micron scale static and dynamic friction can be used to purposefully manipulate soft surfaces to produce well defined frictional gradients.

1. Introduction
The capabilities of atomic force microscopy, AFM, can be exploited in a number of operational modes. Analysis in the force versus distance (F-d) mode is now deployed routinely across a wide range of scientific disciplines in order to measure the local z-component of the force between two objects (usually between tip and surface) at a resolution in the low-pN range, as described in the literature, e.g., [1-3]. Likewise, analysis in the lateral force mode is finding wide application, particularly as a nano-tribological tool e.g., [4-5]. Thus it is recognized that the system is capable of measuring both out-of-plane and in-plane force components. In most cases these operations are carried out in distinctly different operational modes.

The present study demonstrates some of the probe parameters which will influence the nature of static and dynamic properties of the contact condition. We demonstrate that the longitudinal bending mode of the lever is an important factor for consideration when imaging soft elastic surfaces and when stimulated during F-d analysis.

2. Experimental Details
2.1. Materials and SPM Instrumentation
Atomically flat single crystal WTe₂ faces were prepared by cleavage immediately prior to analysis in air. A silicon wafer was also used of sub-nanometer roughness comprising a native oxide layer. Poly(dimethylsiloxane), PDMS (Sylgard®-184) was supplied by Dow Corning as a two part silicone elastomer.

The work was carried out using a multi-technique/multi-mode Scanning Probe Microscope (SPM) instrument: a ThermoMicroscope TMX-2000 Explorer and JEOL JSPM-4200.

The probes were of the beam-shape variety in order to ensure that only the simple lowest-order bending modes contributed to the response.

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3. Results and Discussion

The force-sensing/imposing probe is shown in figure 1. The three familiar expressions for force constants of deformation - arising from simple bending \( (k_N) \), torsion \( (k_T) \) and longitudinal buckling \( (k_L) \) are shown below. The expressions assume the deformation of the lever can be described by the lowest order modes of a long thin beam.

\[
k_N = \frac{3EI}{L^3} \quad k_T = \frac{2EI}{(1+\mu)Lh^2} \quad k_L = \frac{EI}{Lh^2}
\]

where \( E \) is Young’s modulus, \( \mu \) is Poisson’s ratio and \( I \) is the reduced moment of inertia \( \pi h^4/12 \). \( k_T \) and \( k_L \) are stimulated by force components acting along the \( x \)- and \( y \)-directions, respectively, with a coordinate system anchored in the probe as shown in figure 1. \( k_{Tb} \) relates to tip bending.

3.1. Static and dynamic friction in the F-d mode

Hoh and Engel [6] have investigated the effect of lateral forces acting on the tip in the F-d analytical mode. They showed that a downward buckling of the lever will occur during the approach cycle when the tip is in contact with the surface, and conversely an upward buckling in the retract cycle. The result is a differential shift in the retract and approach curves. The tip is attached rigidly at the end of the lever such that the apex of the tip is displaced a vertical distance from the plane of the lever. Therefore stimulation of either the bending or the longitudinal modes will cause displacement not only in the direction of the applied force, but also in the orthogonal direction in the \( y-z \) plane. The \( y \) displacement of the tip apex can be described by the following equation

\[
y = \left( \frac{L \sin \beta \cos \beta}{h} + \left( \frac{L^2 - 3h^2}{3h^2} \right) \sin \beta \cos \beta + 1 \right) \frac{F_N}{k_N} + \left( \frac{L^2 - 3h^2}{L^2} \right) \frac{3h \sin \beta}{L} \cos \beta + \frac{3h}{2L} \frac{F_N}{k_N}
\]

where the \( \sin \) and \( \cos \) terms in the equations take into account the tilt of the tip/lever as being some angle \( \beta \).

The tip apex displacement in the \( y-z \) plane is illustrated by the diagrammatic representation of a generic micro F-d friction loop resulting from contact with an array of stick points (small F-d curve taken while the sample and tip are in contact) as shown in figure 2 (a).

![Figure 1. Schematic of a beam-shaped probe defining the various probe parameters, spring constants and coordinate system.](image)

![Figure 2. (a) The segments ‘a & b’ represent changes in detector response for a given z-stage displacement (i.e., the normal deflection of the lever for an F-d curve without stick-slip responses- approach and retract curves). The tip z-displacement must be measured along the slope of this line. The segment ‘c’ represents the ‘unbuckling’ of the lever when the tip ‘slips’ from one stick point to its nearest neighbour. The distance marked ‘d’ is the displacement of the z-stage corresponding to tip displacement from one stick point to another (i.e., the required z-stage travel to move the tip laterally from one stick-point to the next). The slope of the stick regions (buckling and unbuckling) represents the combined lateral stiffness of the two objects at the point of contact (the lever bending component is also incorporated in this signal. (b) F-d generated friction loops for scanning perpendicular and along the ‘top’ Te rows of WTe2. The scale bars show z-displacement for the stage and tip.](image)
The separation of the approach and retract curves (figure 2) increases (increasing frictional force) with increasing applied load along the z-direction.

Figure 2 (b) shows an example of a micro-F-d loop taken on a WTe$_2$ surface. The response of the lever is monitored on the Top-Bottom (TB) segments of the photo-detector while carrying out Force versus distance curves with the long axis of the lever aligned perpendicular and parallel to the close-packed ‘top’ Te rows (i.e., aligned with the [010] and [100] crystallographic directions, respectively). The ratios of the relative magnitudes of the periodicity and amplitude along the two directions correlate well with the atomic spacings along the two directions on the dichalcogenide surface. The difference in the spacing between the approach and retract curves reflects the difference in dynamic friction for scanning along the different directions on the surface. The friction versus loading can be used to determine the frictional dependence and frictional properties such as friction coefficient and also provides a unique and simple process for z-scanner calibration in the nm range [7-9].

The near-horizontal sections of the discrete steps in the F-d loops represent the TB difference signal for the stationary tip when buckling competes with bending. Figure 3 shows the response of the TB signal for small stage displacements in the z-direction. In figure 3 (a) the F-d curve shows a negative slope (a wholly static friction regime) in the approach and retract curves. The diagram in figure 3 (a) illustrates the absence of lateral movement of the tip apex (i.e., the apex remains trapped at one particular stick-point as the stage/sample is translated through a small F-d cycle). If the lateral force of attachment at the stick position is exceeded by the lever-induced force of buckling, then a slip response will occur and the tip apex will move to the next stable stick position. Thus, the tip will oscillate around stick point/s as the stage completes, and repeats, an F-d cycle (diagram in figure 3 (b)).

Figure 3. (a) & (b) TB detector signals for small stage displacements in the F-d mode on a WTe$_2$ surface with corresponding tip movements. In (a) the tip apex does not move in the x, y plane (ignoring in-plane sample and tip deformation). In (b) the tip apex experiences stick-slip responses.

It should be noted that the lateral deformation of the sample and tip bending will contribute to the slope of the section that represents the tip apex being trapped at a stick point (static friction regime). The lateral and normal deformation of the sample will depend on the force of interaction, and on the force constants of the lever, $k_N$ and $k_L$, and on the effective stiffness of the sample in the z- and y-directions.

The effective lateral stiffness of the system, $k_{eff}$, can be expressed in terms of a series combination of springs.

$$k_{eff} = \left(\frac{1}{k_L} + \frac{1}{k_{TB}} + \frac{1}{k_{contact}}\right)^{-1}$$

(2)
The effect of lateral stiffness will result in a reduced slope for the static friction component - for example on a cadmium arachidate film in comparison with that of a WTe$_2$ surface (for similar loading force). The Langmuir-Blodgett film will experience a greater in-plane deformation than the WTe$_2$ surface.

The influence on tip bending contributions to the static friction component of the F-d loop is shown in figure 4. Significant tip bending during the stick-slip process results in a reduction in the stick response gradient (i.e., the buckling signal is reduced as tip bending along the y-axis is increased). The ‘standard’ (figure 4) F-d curves demonstrates the decrease in adhesion from a smaller tip-sample contact. The smaller F-d loops show at similar force loadings the dynamic friction is reduced (measured by the separation of the approach and retract cycles) when the tip radius is also reduced (45 to 15 nm).

The influence of the longitudinal restoring force of the lever is also demonstrated in figure 5 where ‘standard’ imaging has been carried out on an elastic polymer sample Poly(dimethylsiloxane), (PDMS). Figure 5 (a) shows a topographical image of a PDMS surface after it has been manipulated previously with moderately high force loadings (> 500 nN) under normal raster scanning conditions. The image shows a series of discrete troughs aligned along the fast scan direction of the lever. This phenomena is the result of stick-slip responses of the lever in the slow scan direction during the manipulation process of scanning. Thus at the start of the raster, the tip travels across the surface of the polymer in the fast scan direction and will cause some polymer bond breakage, as well as plastic and elastic deformations. The tip remains in the channel during successive scans and displaces more polymeric material as the scanning continues, while being deflected in the direction of the slow scan, until a lateral force causes escape from the stick-region, and allows the next point of tip-polymer stability to be established. That is, the effective spring constant of lateral interaction exceeds the effective in-plane spring constant of the lever, when the tip is trapped, while the reverse is the case when it escapes in a discontinuous manner.

When probes are utilised with relatively large radii of curvature the distribution of force loading on the polymer surface results in reduced pinning of the contact. Lower force loadings also result in minimal tip trapping along the x and y directions on the polymer resulting in a more homogenous manipulation of the surface. Consequently the surface can be manipulated accordingly as shown in figure 5 (b) producing well defined frictional patterning by alteration of loading forces.

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