Small-Amplitude Atomic Force Microscopy**

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Small amplitude Atomic Force Microscopy (AFM) is a relatively new AFM technique which was specifically developed to perform linear measurements of nanomechanical phenomena. This is achieved by using ultra-small cantilever amplitudes and very high sensitivity deflection sensors. Recently this technique has been used in ultra-high vacuum (UHV) and liquid environments to measure atomic and molecular forces and dynamics with high precision. Here we focus on three examples which are interesting from a nanoengineering standpoint: Atomic energy dissipation (atomic friction), atomic-scale contact mechanics, and nanotribology/molecular ordering in confined liquid films.

1. Introduction

The future of nanotechnology depends not only on our inventiveness, but also on the detailed understanding of the electronics and mechanics of nanoscale, molecular and atomic systems. Scanning Probe Microscopy (SPM) epitomizes nanotechnology as does no other measurement technique, because it is the only technique that can directly manipulate, image and measure properties of nanoscale systems. Atomic Force Microscopy (AFM), probably the most used type of SPM, was invented in 1986 to provide nanometer resolution imaging of insulating as well as conducting surfaces.[1] Shortly after its invention, it was realized that AFM can also be used to measure surface forces as a function of distance from the surface.[2–6]

The basic idea behind AFM is quite simple. A sharp tip attached to a cantilever spring is brought close to a surface. The force between the tip and the surface bends the spring. The deflection of the cantilever is translated into a force via Hooke's law. By scanning the tip over a surface and using a feedback loop to keep the cantilever deflection (force) constant, a topographic image of the surface can be obtained. The most common AFM technique, called 'contact AFM', uses this principle.

The main problem with contact AFM is that it is a static measurement. Consequently, it is prone to drift and low signal-to-noise. To increase the signal, low stiffness cantilevers are used. However, such cantilevers are too weak to resist strong attractive forces, leading to a 'snap-in' instability close to the surface.[6] Thus imaging is done in contact. While this is acceptable for low resolution imaging (~ 10 nm), it is not useful for either atomic resolution imaging or the measurement of force interactions.

To remedy this situation different *dynamic* modes of AFM have been developed.[3,7,8] Here the cantilever is oscillated and the amplitude, phase or resonance frequency are measured. This can be done with better signal-to-noise (using a lock-in amplifier or a frequency measurement) and stiffer levers can be used. Measurements closer to the surface without contact (non-contact AFM)[3] or with intermittent contact ('tapping mode' AFM)[9] become possible, improving imaging resolution.

The majority of dynamic AFM techniques involve oscillating the cantilever at or close to resonance.[7,8] As the tip approaches the surface, a change in the effective compliance of the cantilever results in a change of its resonance frequency. If the driving frequency of the cantilever is kept constant, the amplitude or phase will vary due to this interaction. Using a feedback controller the amplitude or phase are kept constant at a chosen set-point to obtain an image.[3] This mode of operation is called amplitude modulated (AM) AFM. The other dynamic mode of operation is frequency modulation (FM) AFM[8,10] wherein the cantilever is oscillated always at resonance by frequency tracking and measuring the shift from the free resonance frequency. The imaging is performed at constant frequency shift as the tip is scanned across the surface. FM AFM has been found to have superior signal-to-noise and thus allows for the use of very stiff cantilevers. Consequently, imaging can be performed at very close proximity to the surface and short-range chemical bonding forces determine the image contrast. Atomic resolution can be achieved that is comparable to Scanning Tunneling Microscopy (STM).[11] One downside is that FM AFM has to be per-

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formed in ultra-high vacuum (UHV) due to the need of a high quality factor, although some progress has recently been made to use FM AFM in more dissipative environments.[12,13]

There is a downside to most dynamic techniques. The levers are oscillated at resonance and the resulting amplitudes are quite large (of the order of 10–100 nm). Since the cantilever is moving in a non-linear force field, the cantilever-surface system comprises a highly non-linear oscillator.[7,14–18] In addition, interactions are not entirely conservative and even in a clean ultra-high vacuum environment the cantilever will dissipate energy due to interactions with the surface.[19–21] Thus we have a large amplitude oscillator moving in a non-linear force field with non-linear, distance-dependent damping. This has made quantitative interpretation of force data very difficult in many cases. For FM AFM, methods have been found to solve the resulting non-linear equations which range from integral equations, perturbation theory, matrix methods to fractional calculus. Most of these methods, however, assume a conservative force field,[22–25] although some attempts have been made to include dissipative effects.[26–28]

Small-amplitude AFM[29] was invented to circumvent these problems. The basic idea is very simple: Keep the advantages of dynamic AFM, but deliberately reduce the amplitudes to render the measurement as linear as possible. This means in practice that the amplitudes have to be reduced to a fraction of the interaction range, which often means to less than 1 Å.[24,30] Such small amplitudes can easiest be achieved if the cantilever is oscillated far below its first resonance. This has the added advantage that the phase will be zero as long as the cantilever does not experience any damping. Any phase difference will be a direct result of dissipative processes. Thus both conservative and dissipative force terms can be neatly separated from amplitude and phase measurements. Recently, it has been pointed out that using such small amplitudes have an added advantage in that the tip will spend most of its time in the short-range, chemical bonding force field, while at large amplitudes the tip will spend most of its time in the long-range force field, which does not provide atomic resolution.[31,32] Thus as long as stable imaging with a small amplitude can be achieved, the signal will be dominated by short-range interactions and atomic contrast is improved.

Since first experiments with small amplitude AFM in 1993,[33] this technique has been successfully used to measure chemical bonding forces,[34,35] few-atom contact mechanics,[20,35] atomic-scale dissipation,[20] and to achieve atomic[36] and sub-atomic[11] resolution in ultra-high vacuum, as well as to investigate molecular ordering[37] and nanotribology in liquids, including water.[38]

2. Small Amplitude AFM

As discussed above, small amplitude AFM is any dynamic AFM technique in which amplitudes smaller than the measured interaction range are used. In practice, four different modes have been successfully used in the past (in chronological order):

1. *Force-modulated AFM*:[33,34,37] In this mode a small oscillatory force is applied directly to the end of the cantilever. This is typically done by attaching a magnetic particle to the cantilever and applying a small AC magnetic field to excite the lever. The excitation frequency can be at or below the resonance. In addition, a feedback-controlled DC field can be applied to stabilize the cantilever.

2. *Resonance AM AFM*:[39,40] In this mode (as in Modes 3 & 4), the cantilever is oscillated via the more common method of applying a *displacement* to its base (via a piezoelectric actuator) rather than a force. The cantilever is oscillated at its free resonance and the amplitude is measured. This is similar to regular non-contact and tapping mode with the exception that amplitudes are small and levers are very stiff to avoid snap-in.

3. *Sub-resonance AM AFM*:[20,35,36,38,41,42] The cantilever is oscillated at a frequency far below its first resonance and the amplitude and phase are monitored. This has the advantages of making it easy to maintain small amplitudes and allowing easy separation of conservative and dissipative forces.

4. *FM AFM*:[11,21,32,43] Recently, the advantages of FM AFM at small amplitudes have been stressed and several groups are focusing on developing this technique.

The data interpretation depends on the particular mode used, but in each case the technique measures the gradient of the force or interaction stiffness. Also in each case, the force gradient (and the damping coefficient) can be obtained from simple algebraic equations, due to the in inherent linearity of the technique. As an example, we will focus on sub-resonance AM AFM (Mode 3).

The general equation of motion of cantilever & tip interacting with the surface can be written as:[29,30]

$$m^* \frac{d^2 z}{dt^2} + C(z + z_0)\frac{dz}{dt} + k_L z = k_L A_0 \exp(i\omega t) + F(z + z_0) \qquad (1)$$

Here, z is the deflection of the cantilever, z_0 is the offset of the tip, m^* is the effective mass of the moving cantilever plus tip, C is the distance dependent damping coefficient, k_L is the cantilever stiffness, A_0 is the drive amplitude, and F is the force between tip and surface. At small amplitudes the force can be linearized to $F(z+z_0) = F(z_0) + k_i z$ and the damping can be considered constant over the motion of the cantilever. Here k_i is the interaction stiffness, which is the negative of the force gradient

$$k_i = -{}^{dF}/_{dz} \qquad \text{formula (1)}$$

We can rescale the distance axis by replacing $z+z_0$ with z, which then becomes the deviation of the cantilever from its equilibrium position in the force field, i.e. the amplitude. We obtain:

$$m^* \frac{d^2 z}{dt^2} + C\frac{dz}{dt} + (k_L + k_i)z = k_L A_0 \exp(i\omega t) \qquad (2)$$

This is the equation of a linear forced oscillator with the well-known solutions:

$$|A| = \frac{k_L A_0}{\sqrt{\left(k_L + k_i\right)^2 \left(1 - \frac{\omega^2}{\omega_0^2}\right)^2 + (C\omega)^2}} \tag{3}$$

and

$$\tan\phi = -\frac{\omega C}{\left(k_L + k_i\right)\left(1 - \frac{\omega^2}{\omega_0^2}\right)} \tag{4}$$

Here |A| is the absolute value of the amplitude, ϕ is the phase angle between the drive (typically a piezo crystal) and the lever end, and ω_0 is the resonance frequency of the cantilever given by:

$$\omega_0 = \sqrt{\frac{k_L + k_i}{m*}} \tag{5}$$

Equations 3–5 are all that is needed to calculate the interaction stiffness and damping coefficient for techniques 2-4 mentioned above. (In technique 1, a force is imposed on the cantilever instead of a displacement as in cases 2–4. This leads to very simple although slightly different equations.)

In the particular case of sub-resonance operation (Mode 3) the term $1-\omega^2/\omega_0{}^2$ becomes unity and we can solve for k_i and C to obtain:

$$k_i = k_L\left(\frac{A_0}{|A|}\cos\phi - 1\right) \qquad C = \frac{k_L A_0}{A\omega}\sin\phi \tag{6a,b}$$

A typical force measurement therefore consists of an initial measurement of the free amplitude (A_0) and subsequent monitoring (by using a lock-in amplifier) of the amplitude $|A|$ and the phase ϕ as the tip approaches the surface.

3. Experimental

In small amplitude AFM, three issues become especially important: sensitivity, stability and noise. Since very small amplitudes are used, signal-to-noise will be diminished. Thus it is important to use a very sensitive deflection sensor and to reduce noise as much as possible. Another effect of small amplitudes is the reduced stability. Large amplitude techniques have the advantage that the restoring force of the cantilever is often large enough to avoid snap-in. In small amplitude techniques, the restoring force tends to be small since the deflection is small. Thus instabilities have to be avoided by using very high stiffness cantilevers. In UHV, this should be at least 100 N/m. The use of such high stiffness cantilevers reduces the signal even further, making a high sensitivity deflection sensor crucial. Common AFM use laser deflection systems, which typically cannot measure amplitude changes smaller than 1 Å. This is not sufficient.

One possibility for a very high sensitivity deflection sensor is a fiber-based interferometer[41,44] which has been shown to give at least a hundred-fold increase in sensitivity over conventional AFM. Fiber interferometers essentially operate as Fabry-Pérot interferometers where the cavity is formed by the fiber end and the back of the cantilever. By using semi-reflective coatings on the fiber end and a high-precision alignment mechanism, these systems can measure amplitude changes as small as 0.01 Å, one hundredths the width of a single hydrogen atom.

At such small amplitudes the question arises if the measured amplitudes are beyond the thermal noise of the cantilever. Most of the thermal noise will be concentrated in the resonance peak. Thus another advantage of sub-resonance operation is the reduction of thermal noise. With a 100 N/m cantilever and a Q of 1000, the thermal noise at sub-resonance frequencies is lower than 10^{-4} Å at room temperature, in contrast to on-resonance where the thermal noise will be closer to 1 Å.[29] Consequently, much stiffer levers must be used in small-amplitude FM AFM (> 1000 N/m).[43]

4. Results

Small amplitude AFM has been used for linear measurements and high resolution imaging (see Fig. 1) in a variety of application where quantitative measurements are important. These include atomic and subatomic imaging,[11,36] forces between single atoms,[35] atomic energy dissipation,[20,21] contact mechanics of few atom contacts[20,35] as well as molecular ordering, dissipation, and nanotribology in liquids.[37,38] Here we want to focus on three examples which might be of most interest from an engineering stand-point: Atomic energy dissipation & friction, few atom contacts, and nanotribology in confined liquids.

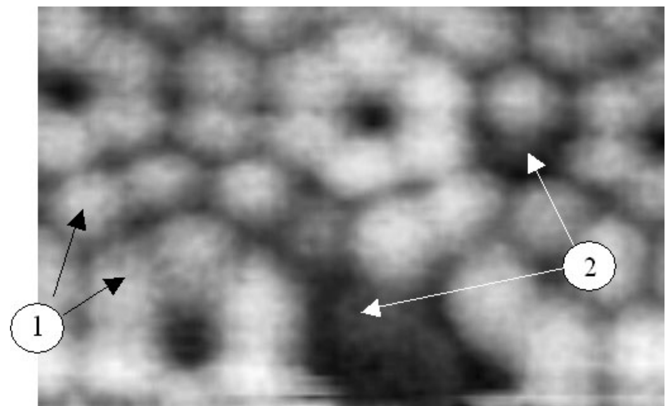

Fig. 1. Atomic resolution force gradient image of Si(111) 7×7 obtained with a small amplitude AFM/STM. Free amplitude A^0 was 0.25 Å. Arrows 1 point at adatoms and arrows 2 point at defect sites and rest atoms visible in the exposed underlayer.[36]

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4.1. Atomic Friction

As the cantilever oscillates up and down during a measurement it is found to lose energy due to a variety of damping processes. In air and liquids a large part of the dissipated energy is due to the motion of the cantilever through a medium. In UHV, in the absence of a surrounding medium, energy dissipation is solely due to internal friction of the cantilever and any dissipative interactions with the surface. Recently it was found that in dynamic AFM in UHV, there is a distance-dependent energy loss close to the surface. This loss is typically of the order of 0.01–1 eV per cycle. Figure 2[20] shows a simultaneous measurement of the interaction stiffness and the energy dissipation measured with a Pt-Ir tip on a Cu(100) surface. It can be seen that the energy dissipation onset is coincident with the appearance of a short-range atomic interaction.

This dissipation can be modeled if we assume that there is an atomic defect that can move between two mechanical states with an activation energy of about 0.44 eV. When the tip moves towards the surface, the defect is in its stable state. The force interaction with the tip causes the defect to 'flip' into a different state, and thus the tip experiences a different force field on its way up. When the tip is far enough away the defect relaxes back to its original state. The resulting mechanical hysteresis is responsible for the observed energy dissipation. This opens up the possibility to explore atomic, molecular and nanoscale friction phenomena in a quantitative manner. Understanding dissipative processes at the nanoscale is crucial to the development of Nanometer Electromechanical Systems (NEMS) and future molecular and atomic scale devices.

4.2. Few Atom Contacts

Another area of potential interest in the design of future NEMS devices is how far continuum mechanics can be applied as critical dimensions shrink to only a few atoms across. To explore this question, contact measurements between a tungsten sharp tip and a Si(111) surface were performed in UHV and the data was analyzed using continuum contact mechanics.[35] Interestingly, while discrete atomic size jumps were observed in the data, continuum mechanics could be applied to the overall shape of the stiffness curve and reasonable values for the interfacial energy could be extracted. Figure 3 shows a stiffness measurement of few atom contacts between a sharp tungsten tip and a clean silicon surface in UHV. The stiffness was modeled using JKR-DMT theory. There was only one independent adjustable parameter (the interfacial energy γ) and a high confidence fit could be obtained. The best fit was obtained for γ = 1.9 J/m^2 and a tip radius of 1 nm. The fit also allowed the researchers to calculate the yield strength (7–10 GPa) and the elastic energy per atom at yield (0.2–0.6 eV). Both these numbers are very reasonable. The fact that continuum mechanics can be applied to this system is remarkable, considering that the number of atoms in the contact was estimated to be 25–65 atoms. Systems in this size range seem to be at the boundary where the continuum picture still applies but is beginning to break down.

4.3. Nanotribology

Sub-resonance small amplitude AFM is a very versatile technique. Unlike FM AFM a high quality factor is not crucial and it can be just as easily used in liquids. Sub-resonance AFM has been used to measure forces arising from the confinement of various liquids.[37,38,45] With some modifications to the cantilever to decrease its torsional stiffness, lateral measurements become also possible. When liquids are confined between two surfaces, they tend to layer along the surfaces

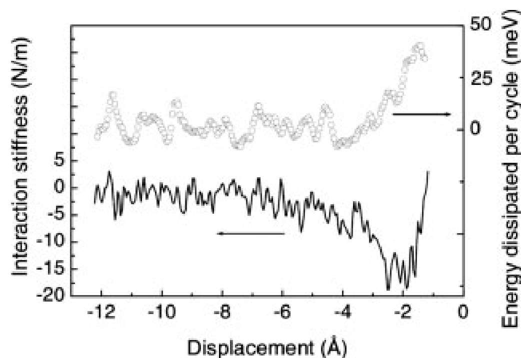

Fig. 2. Interaction stiffness and mechanical energy dissipation determined from a simultaneous measurement of amplitude and phase using sub-resonance, small amplitude AFM. The free amplitude was 0.13 Å. The onset of the energy dissipation coincides with the onset of short-range interactions. The dissipation can be modeled by assuming that there is an atomic scale friction mechanism which involves an atom moving between two states with an activation energy of 0.44 eV.[20]

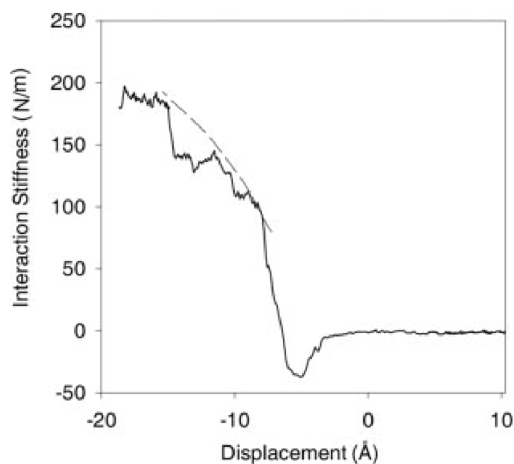

Fig. 3. Stiffness versus displacement for a tungsten tip approaching a clean silicon surface in ultra-high vacuum. Between 0 and –10 Å there is a small attractive region associated with chemical bonding and van-der-Waals forces. Between –10 to –20 Å the tip contacts the surface and there are atomic scale mechanical instabilities as the tip indents the surface. However, a continuum contact mechanics model fits the overall envelope quite well, although only about 25–65 atoms are involved in the contact.

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leading to the development of 'order' normal to the surface. This order can be enhanced if the two surfaces are spaced such that the molecules can neatly order in between. However, if the spacing is not an integer of the molecular spacing, the molecules can not properly pack in the space between the surfaces and ordering is frustrated. In the ordered case, the stiffness of the confined liquid film is often seen to be increased beyond the bulk value, while in the disordered, frustrated case the stiffness is less than the bulk value. This is shown in Figure 4, where the stiffness of a confined water layer was measured with a lever amplitude of 0.35 Å.

By combining lateral (shear) measurements the influence of normal ordering on shear and friction can be explored. Recently measurements on a silicone oil (OMCTS) showed that the lateral stiffness is indeed enhanced to 3-4 times its bulk value when the liquid is allowed to order. However, in the disordered state, the shear stiffness remains low even when the load on the liquid film is already substantial. These findings are expected to have important implications for friction in nanomechanical devices.

5. Summary and Outlook

In summary, small-amplitude AFM has made tremendous progress in less than 10 years to become one of the main *quantitative* measurement techniques in the area of nanomechanics. Questions of the limits of continuum mechanics, atomic (dry) friction and nanotribology in liquids are becoming increasingly important for the design of future nano-electromechanical devices. We have found that even for systems as small as 25 atoms, the continuum approach still gives meaningful results. However, we have also seen that this approach begins to break down for systems of that size. The limit of the continuum approach has to be determined on a case-by-case basis by using techniques such as small-amplitude AFM. In cases where continuum mechanics is no longer applicable, the emerging properties of the nanoscale system have to be explored. We expect that small-amplitude AFM will continue to be one of the major techniques to investigate these issues.

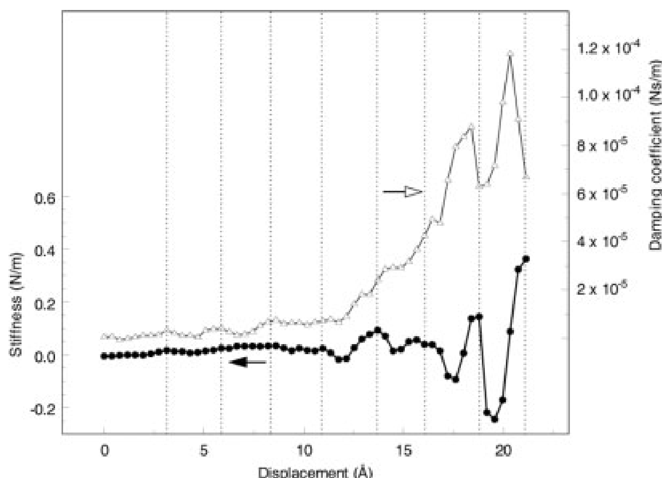

Fig. 4. Measurement of the stiffness and damping coefficient of a water film confined between an AFM tip and a mica surface. The stiffness oscillates because of confinement induced order or disorder, depending on the tip-surface separation. The vertical damping coefficient also shows oscillatory behavior, suggesting that the ordering of the liquid affects the energy dissipation in the system.[38]

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