

Intermittent-Contact Mode Force Microscopy & Electrostatic Force Microscopy (EFM)

Table of Contents:

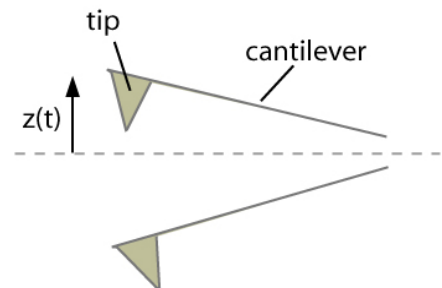
1. Motivation	1
2. Simple Harmonic Motion	1
3. AC-Mode Imaging	3
4. Electrostatic Force Microscopy	4
5. Applications.....	5
References.....	5

1. Motivation

Contact mode, non-contact mode, and intermittent-contact mode scanning probe microscopy (SPM) all use short range van der Waals forces to probe the topographic features of a sample. However, if we move the scanning probe further away from the surface (10-50 nm), we can examine longer range forces, such as electrostatic and magnetic forces. In doing so, we can learn about the electrical properties of a material with high spatial resolution. Electrostatic force microscopy (EFM) and its variants are used to study topics as diverse as dopant profiles in silicon transistors, charge injection in organic diodes, and changes in photosensitive reaction complexes in biological membranes. Both intermittent-contact mode and EFM make use of the changes of the motion of an oscillating tip to produce images. In order to understand these imaging modes we first need to review the motion of an oscillating tip. If you have studied simple harmonic motion in your physics or math classes, the next section may be familiar to you.

2. Simple Harmonic Motion

The SPM tip sits at the end of a long, flexible cantilever. This cantilever is flexible and behaves like a spring: if the tip is pushed in one direction the cantilever exerts a force in the opposite direction in an attempt to restore the tip to its original position. Since we are going to be examining the motion of the tip in more detail, we first define some parameters:



- $z(t)$ position of the tip as a function of time
- F force exerted on the tip
- m mass of the tip
- k effective spring constant of the cantilever

Remember that the velocity $v(t)$ and acceleration $a(t)$ of the tip are related to its position $z(t)$ through the following derivatives:

$$v(t) = \frac{dz}{dt} \qquad a(t) = \frac{dv}{dt} = \frac{d^2z}{dt^2}$$

Newton's third law ($F = ma$) relates the forces on the tip to its motion. We already mentioned that the cantilever behaves much like a spring, and will we approximate the restoring force using Hooke's law relating spring constants and restoring forces ($F = -kz$). Combining these two equations gives us the following equation of motion:

$$F = ma = -kz$$

$$m \frac{d^2z}{dt^2} = -kz$$

The tip and cantilever are real materials moving through air, so the motion is also damped by both air friction and by losses in the spring. These losses are both approximately proportional to the velocity and so we modify our equation of motion with a "drag force" or damping term $-bv$:

$$F = ma = -kz - bv$$

$$m \frac{d^2z}{dt^2} = -kz - b \frac{dz}{dt}$$

Rearranging a bit, we can write this as a differential equation describing basic motion of the tip far away from any substrate (and still without any driving force yet either)

$$m \frac{d^2z}{dt^2} + b \frac{dz}{dt} + kz = 0$$

$$\frac{d^2z}{dt^2} + \beta \frac{dz}{dt} + \omega_0^2 z = 0$$

where $\omega_0^2 \equiv k/m$ and $\beta \equiv b/m$. ω_0 is the natural frequency of the cantilever.

Most non-contact/intermittent contact SPM is performed while applying a sinusoidally oscillating drive force on the tip, so we must also add the driving force to our equation of motion:

$$\frac{d^2z}{dt^2} + \beta \frac{dz}{dt} + \omega_0^2 z = D \cos \omega t$$

This equation may look familiar. It is the classic equation for the damped and driven simple harmonic oscillator. The solution to this equation is a steady-state motion of the system should oscillate with the driving force, plus a potential phase shift δ . You can check that the solution has the form:

$$z(t) = A \cos(\omega t - \delta)$$

where:

$$\delta = \tan^{-1} \left(\frac{\omega\beta}{\omega_0^2 - \omega^2} \right)$$

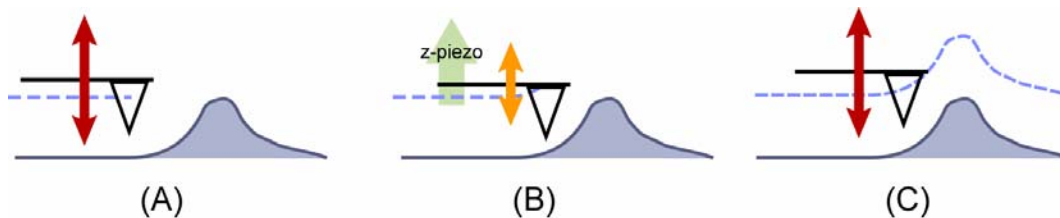
The amplitude, A , of oscillation is:

$$A = \frac{D}{\sqrt{(\omega_0^2 - \omega^2)^2 + \omega^2 \beta^2}}$$

The “resonance frequency” of the oscillator is defined as the driving frequency at which A is maximized. If the damping is small, the amplitude is at a maximum when the driving frequency equals the natural frequency ω_0 . The amount of damping in a simple harmonic oscillator is commonly characterized by the quality factor, Q . Q is defined as the resonance frequency divided by β , and is a measure of the total energy stored in the oscillator divided by the energy lost per period of oscillation. For systems that are only weakly damped (like an AFM tip vibrating in air), a practical method of determining Q is to divide the resonance frequency by the width of the resonance peak (where the width is taken at the points where the amplitude is equal to $1/\sqrt{2} \approx 0.707$ of the maximum).

3. AC-Mode Imaging

In a common form of topography imaging, called intermittent-contact mode (or a variation called “Tapping Mode™” by some manufacturers), the AFM is driven at a frequency close to the resonant frequency of the tip. When the tip comes close to the surface, it interacts with the surface through short range forces such as van der Waals forces. These additional interactions change the resonance frequency of the tip, thereby changing the amplitude of oscillation and its phase lag. Typically, an image is formed by using a feedback loop to keep the oscillation amplitude constant by varying the tip sample distance with the z-piezo (see figure below). By plotting the z-piezo signal as a function of position it is then possible to generate an image of the height of features on the surface. It is possible to image in both the attractive, and the repulsive regions of the van der Waals potential, and strictly speaking this divides the classification of AC-Mode imaging techniques into “non-contact” and “intermittent contact” AFM respectively). However, intermittent contact AC-mode imaging is more common for routine imaging.



Schematic for intermittent-contact mode AFM. (A) The AFM tip is driven near its natural resonance frequency to obtain a target amplitude of oscillation. (B) As the tip approaches changes in topography, the increasing van der Waals forces shift the resonance frequency, which causes the tip’s oscillation amplitude to decrease. In response, the Z-piezo lifts the tip away from the surface so (C) the original oscillation amplitude is reestablished. By tracking the Z-motion of the tip, we obtain the measured topography of the surface (dashed line).

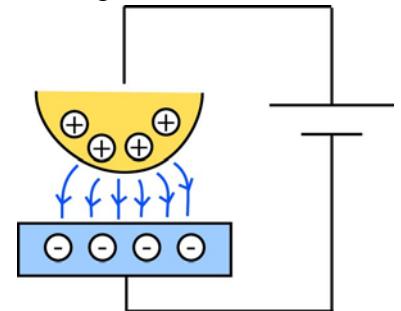
Of course, the material properties of the sample affect tip-surface interactions, so the topography image obtained by AC-mode imaging isn't perfectly free of artifacts (some imaging modes even exploit differences in elastic properties of the surface to differentiate materials). Nevertheless, AC-mode imaging is much gentler and can be used to image a wider variety of soft samples than contact mode imaging.

4. Electrostatic Force Microscopy

The goal of an EFM experiment is not to image the surface topography, but rather to image the electrical properties of the surface. This is accomplished by vibrating a metal-coated tip some distance away from the surface — beyond the range of short range van der Waals interactions, so that only long-range forces (such as electrostatic forces) remain.

As the tip scans over a surface, its motion is affected by the forces between it and the surface below. To understand EFM, it is useful to think of the tip and sample as forming two plates in a very small capacitor. The potential energy, U , of a capacitor with capacitance C charged to a voltage V is given by:

$$U = \frac{1}{2} CV^2$$



The force F , is always given by the first derivative of the potential U , as a function of position z . The force between the tip and the cantilever is then:

$$F = -\frac{dU}{dz} = -\frac{d}{dz} \left(\frac{1}{2} CV^2 \right) = -\frac{1}{2} \frac{dC}{dz} V^2$$

This force depends on both the applied voltage (independent of distance) and the capacitance (depends on distance). This is really an electrostatic force (Coulomb's Law) in a different form. The potential difference causes opposite charges to build up on the tip and the substrate (as seen in the cartoon), which pulls the tip towards the substrate.

How does this force affect the tip motion? Well, to first order it doesn't! (You may remember from your physics coursework that the resonant frequency of a spring-mass oscillator doesn't depend on the value of the local gravitational acceleration). It is not the electrostatic force, but rather the *force gradient* that determines the oscillation of the tip. For example, the force gradient using the restoring force from the cantilever (Hooke's Law) is

$$F = -kz$$

$$\frac{dF}{dz} = -k$$

This is the spring constant, which shows up in the natural frequency ω_0 of the oscillator. Any other forces on the cantilever that have a significant force gradient will affect the tip's motion. In this case, we are interested in measuring the electrostatic force by how

it affects our oscillating tip. It is easier to see the effect of this additional force on the tip's motion by writing its contribution to the total force gradient:

$$F = -kz - \frac{1}{2} \frac{dC}{dz} V^2$$

$$\frac{dF}{dz} = -k - \frac{1}{2} \frac{d^2C}{dz^2} V^2$$

Assuming $\frac{d^2C}{dz^2}$ is relatively constant for the range of motion, we can consider this new factor as a change in the spring constant, $k' = k + \Delta k$. **In other words, the electrostatic force gradient works like a change in the effective spring constant of the cantilever.** This will change the natural oscillation frequency, now ω_0' , and the phase shift δ' between the cantilever and drive signal for some constant driving frequency ω .

$$\omega_0' = \sqrt{\frac{k'}{m}}$$

$$\delta' = \arctan\left(\frac{\omega\beta}{\omega_0'^2 - \omega^2}\right)$$

For small damping factors and a driving frequency at the resonance frequency ($\omega_0 = \omega$), we can approximate the change in phase shift as [3]:

$$\Delta\delta = -\arcsin\left(\frac{Q}{2k} \frac{d^2C}{dz^2} V^2\right)$$

In real experiments, the phase shift versus voltage curve has a non-zero offset, V_0 :

$$\Delta\delta = -\arcsin\left(\frac{Q}{2k} \frac{d^2C}{dz^2} (V - V_0)^2\right)$$

This offset is attributed to the difference in work function between the tip and the sample.

5. Applications

Electrostatic force microscopy and variations of EFM (such as scanning Kelvin probe microscopy (SKPM), measuring differences in surface potential; magnetic force microscopy (MFM), measuring magnetic moments) can be used to characterize many different samples, such as dopant profiles in silicon-based transistors, polarization in piezoelectric materials, and charge distributions in organic semiconductors. In this lab, you apply EFM to characterize metal films as well as semiconducting polymer blends.

References

1. *Scanning Probe Microscopy and Spectroscopy: Techniques, and Applications*. Edited by Dawn Bonnell, Wiley-Interscience Publication, 2nd Ed., New York, 2001.
2. *Classical Dynamics of Particles and Systems*. Jerry B. Marion and Stephen T. Thornton, Harcourt Brace & Company, 4th Edition, 1995.
3. C H Lei, A Das, M Elliot, J E Macdonald, "Quantitative electrostatic force microscopy-phase measurements." *Nanotechnology*. **15** (2004) 627-634.