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AFM Tutorial

Within the past decade, a family of powerful surface imaging techniques, known collectively as scanned probe microscopy (SPM), has developed in the wake of the invention of the scanning tunneling microscope (STM). Each scanned probe technique relies on a very sharp probe positioned within a few nanometers above the surface of interest. Some combination of probe and/or substrate positioning is required to provide sub-nm-resolution, three-dimensional motion of the probe relative to the substrate. When the probe translates laterally (horizontally) relative to the sample, any change in the height of the surface causes the detected probe signal to change. In general, if the probe signal decreases, this means that the point on the surface directly beneath the probe is farther from the probe than the previous point was. Conversely, if the probe signal increases, then the point on the surface is closer to the probe than the previous point.

The electronic circuit that controls the **vertical** position of the probe relative to the sample uses these changes in the probe signal as sensory feedback to decide which direction (up or down) to move the probe to maintain a constant probe signal. When the probe signal decreases, the circuit realizes that the surface is now farther away, so it moves the probe down until the signal increases to the same level that was measured at the previous point. Similarly, the circuit responds to increases in probe signal by moving the probe up, away from the surface, until the signal decreases back to the desired level. The distance that the probe is moved up or down to return the probe signal to the desired value is therefore related to the height at each point.

Figure 1. General principles of a scanned probe microscope.

To create a 3-dimensional map of surface height, the probe is scanned horizontally (let's say parallel to the x axis) along a series of parallel lines, while the height at each point along each line is recorded. Each scan line is displaced laterally from the previous line by a small distance along the y axis, so that, after several hundred lines have been scanned, the total displacement in the y direction is equal to the length of each line in the x direction, and a square region of the sample has been measured. We now have a measure of height at each point in a 2-dimensional region of the sample, or z(x,y), which is exactly analogous to a topographic map of (if the surface is very rough, for example) the Grand Canyon, except that the dimensions are scaled down by a factor of a trillion (1012) or so!

Probe signals that have been used to sense surfaces include electron tunneling current, interatomic forces, photons, capacitive coupling, electrostatic force, magnetic force, and frictional force. In two prominent cases (STM and atomic force microscopy, AFM), the probe signals depend so strongly on the probe-substrate interaction that changes in substrate height of as little as 0.01 nm can be detected. In addition, the STM and AFM probes can interact with regions of the substrate that are of atomic-scale lateral dimensions, which allows the substrate height to be measured with sub-nm lateral resolution as well. It was the ability of the STM to image individual atoms on surfaces that won the inventors of the STM (Gerd Binnig and Heinie Rohrer of IBM Research in Zurich) the Nobel Prize for Physics in 1986.

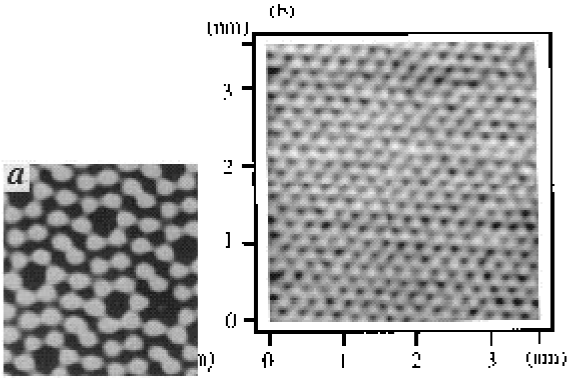


Figure 2. Examples of atomic resolution images acquired with scanned probe microscopes: a.) STM of silicon [Si (111) 7x7 reconstruction; 2x2 nm]; b.) AFM of graphite (Highly Oriented Pyrolytic Graphite; 3.5x3.5 nm).

The Scanning Tunneling Microscope

STM image information is derived from measurements of the electron current that can flow when two electrodes, one a sharp metal tip, and the other a relatively-flat, conducting sample, are brought to within about one nanometer of each other. When the two electrodes are so close together (a few atomic radii), electrons can pass from one electrode to the other by tunneling through the potential energy barrier (think of it as a wall) that normally confines them inside each electrode. Electrically biasing the tip electrode relative to the sample allows more electrons to travel in one direction than in the other, so a net current flow (which can be measured) is established. This is our probe signal, the tunneling current.

The magnitude of the tunneling current is a very strong function of the distance (we'll call it s) between the probe tip and the sample. In fact, the current is so sensitive to the probe/sample spacing that, under normal operating conditions, it changes by about a factor of ten for a change of only 0.1 nm in the separation distance (a distance smaller than the radius of a single atom!). Now think about this: The probe tip is made of atoms, and, if the tip is sharp, chances are good that one of the these atoms sticks out a little farther than the others. Therefore the tunneling barrier is thinnest right below this protruding atom so it is much easier for electrons to tunnel between the tip and the sample at this point than anywhere else. Consequently, the majority of the tunneling current will flow through this single atom, allowing us to sense changes in the sample height with atomic-scale lateral resolution.

In practice, the tip is first approached toward the sample until a tunneling current is detected, at which point a constant current feedback loop is turned on. The feedback circuit responds to changes in the current and varies the voltage applied to the tip-positioning piezoelectric element until the current reaches the desired value (the set current). When the tip is moved laterally to a new position above the sample, the current will change if the tip-to-sample distance changes. The control unit then moves the tip up or down until the current matches the set current, which is equivalent to restoring the tip-to-sample distance to its previous value. Since the tip is moved to the same tip-to-sample distance above each point on the surface, the STM is actually tracing out a replica of the surface topography, as is shown in Figure 3. A record of the voltage applied to control the tip height at each point can therefore be converted into a constant current image of the topography of the surface.

An alternative mode of STM imaging, referred to as the constant height mode, uses the tunneling current directly as a measure of topographic changes. The scan rate of the STM is increased, while the gain of the constant current feedback loop is reduced to the point that the controller cannot respond to the current changes induced by individual features on the surface. The current changes themselves are then recorded as the image information. This mode has the advantage that images can be acquired more quickly, however, current changes do not provide as direct a link to feature heights.

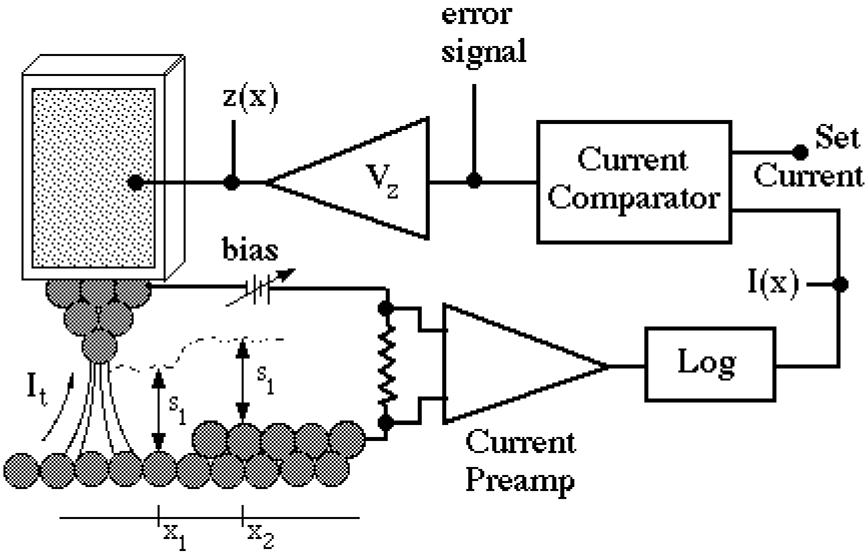


Figure 3 Basic concepts of STM imaging. Tunneling current (Itun) is exponentially dependent on distance between tip and sample (usually ² 1 nm). As the tip is moved from x1 to x2, the current increases as the tip-to-sample distance decreases due to the change in sample height. The increase in current causes the control loop to move the tip away from the sample until the error signal is again zero. Recording the value of the tip height (z) as a function of position (x,y) allows the 3-dimensional topography to be reconstructed.

Problem 1.

The tunneling current at a tip bias of 0.1 volt is found to be given by , where I0 and s0 are constants , and s is the distance between the tip and the sample. For I0 Å10 microamperes, ands0 = 0.0434 nm, plot log10(Itun) vs s , and find the distance between the tip and sample at which the tunneling current is 1 nanoampere. What is the effective electrical resistance of the tunneling barrier when the distance between the tip and sample is 1 nm? (clue: remember Ohm's Law)

THE ATOMIC FORCE MICROSCOPE

Instead of tunneling current, an atomic force microscope (AFM) senses interatomic forces that occur between a probe tip and a substrate. The AFM probe tip, which is similar in purpose to the stylus on a phonograph (you remember phonographs, don't you?), is normally integrated into a microfabricated, thin film cantilever. Once contact between the probe tip and the sample surface has been established, the sample is translated laterally relative to the probe tip, while the vertical position of the cantilever is monitored. Variations in sample height cause the cantilever to deflect up or down, which changes the position sensor output, thus generating the error signal that the feedback circuit uses to maintain a constant cantilever deflection (constant force). Normal imaging forces are in the 1-50 nanonewton range (1 newton is little less than 1/4 pound, after cooking), and cantilever deflections of less than 0.1 nm can be detected.

Detection of cantilever deflection

Given that most of the positioning, vibration isolation, and feedback hardware used for STM can be adapted for use in any scanned probe instrument, the additional instrumental requirement for AFM is only the ability to detect sub-nm deflections of a cantilever. Unlike STM, in which the probe signal is always detected with a preamplifier in series with the tunneling current, the AFM probe signal can be derived from several possible methods for detecting cantilever deflection.

Tunneling sensor:

The first realization of atomic force microscopy relied on an STM tip to establish a tunneling current to the back side of the cantilever so that deflections due to cantilever-substrate forces could be detected.[1] Although a tunneling sensor is very sensitive to cantilever deflection, this method proved to be very inconvenient to implement, and the STM-based sensing technique was quickly superseded by simpler methods.

Optical lever:

First, and still foremost among alternatives to tunneling detection is the optical lever scheme.[2] A laser beam is trained on the back surface of the cantilever, and the reflected beam is sent to a photodiode that is divided into two sections, A and B. Due to the macroscopic length of the reflected light path, any deflection of the cantilever causes a magnified lateral displacement of the reflected laser spot on the photodiode. The relative amplitudes of the signals from the two segments of the photodiode change in response to the motion of the spot-the difference signal (A-B) is very sensitive to cantilever deflection; detection of deflections of <0.1 nm is readily achieved. Optical lever detection is currently used in all but a few commercial AFM instruments.

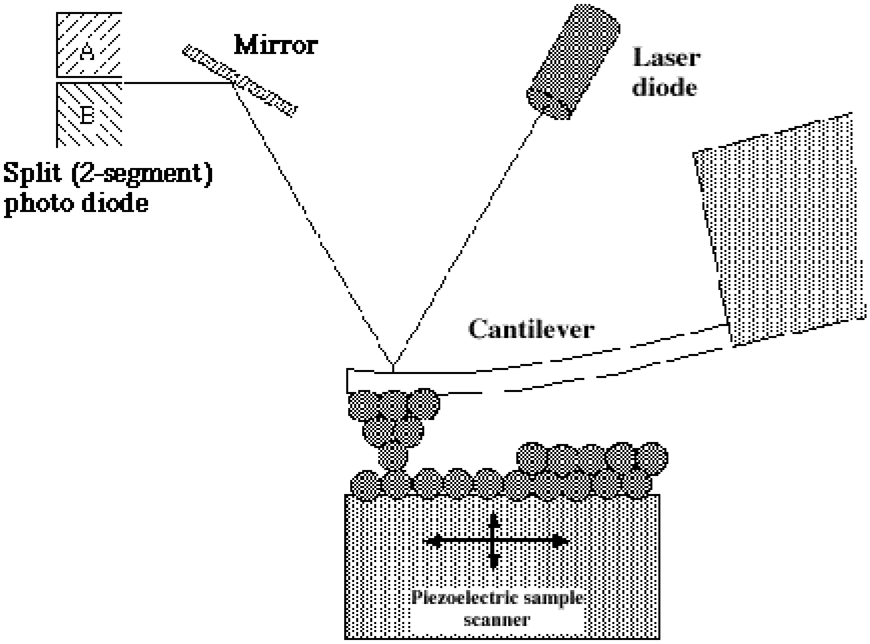


Figure 4. Optical lever detection of cantilever deflection.

Problem 2.

What we normally consider contact is a repulsive interaction between two surfaces-we push on the surface, and it pushes back. This happens only when the distance between the surfaces is very small. Just outside the range at which repulsive interactions become dominant, the force between two surfaces is actually attractive, due to van der Waals interactions. The AFM can sense both types of force. Repulsive forces are defined as positive, and attractive forces as negative. These interactions are often described by using the Lennard-Jones Potential, which gives the potential energy of two atoms separated by a distance r as . Graph U(r)/Umin vs. r for r0 = 0.268 nm. At approximately what value of r is U(r) a minimum (it's negative)? The force between two atoms is defined as . Graph F(r)/Umin vs. r. At exactly what value of r is F(r) = 0? What is U(r)/Umin here?

SPM INSTRUMENTATION

As mentioned above, the standard elements of all scanned probe instruments are primarily derived from those employed for STM. These include:
Figure 5. Piezoelectric tube scanner.

Piezoelectric transducers for sub-nm positioning:

Measuring a surface with sub-nm resolution in three dimensions requires not only a sensitive probe, but also a means of positioning that probe relative to the sample with equivalent spatial resolution. In almost all cases, positioning in scanned probe instruments is accomplished with piezoelectric transducers, usually in the form of cylindrical tubes.3 The application of a voltage between electrodes on the inner and outer surfaces of the tube causes the length of the tube to increase or decrease, depending on the polarity of the bias. If the outer electrode is sectioned into four equally sized electrodes along the length of the tube, then the a voltage applied between the inner electrode and one of the outer electrodes causes the tube to bend. The application of complementary biases (positive and negative) to two electrodes on opposite sides of the tube effectively doubles the magnitude of the bending. Biases applied to the other two electrodes (those 90¡ from the first two) induces bending orthogonal to the previous mode. These two bending modes are used for lateral displacements of the probe relative to the sample.
The extent of the displacement is approximately proportional to the applied bias and the length of the tube. Vertical displacements are imposed by changing the bias of the inner electrode relative to all four of the outer electrodes. The response of the transducers is usually in the range of 1-400 nm of lateral (bending) motion for each volt applied, which gives corresponding maximum lateral scan ranges of from <1 µm to Å160 µm for conventional instrumentation based on high voltage op amps with ±220 volts output (e.g., Apex PA85). The

change in tube length per volt is usually 2-10 times less than the lateral motion, the disparity increasing with increasing tube length. Maximum motions along the tube axis fall in the range of <1 µm to Å15 µm, again, depending on the tube length. The tubes themselves are most often machined from PZT-5A (a lead zirconium titanate material), and range in size from 3-20 mm in diameter, and 10-50 mm in length, with a wall thickness of 0.5-1 mm.

Although tubes are the overwhelming choice for SPM applications, other piezoelectric transducer configurations have been demonstrated. Early STM's were based on three orthogonal piezoelectric bars that each provided motion along only one axis. The orthogonal bar arrangement was rapidly superseded because of the superior range and frequency response characteristics of tubes.

Probe-position-control feedback:

Scanned probe measurements of surface topography are usually based on individual traces obtained by scanning the probe laterally relative to the surface while simultaneously moving the probe (or sample) up or down to maintain a constant probe signal (e.g., tunneling current, cantilever deflection). The up and down motion that allows the probe to follow the contour of the surface is generated by a feedback circuit that is based on simple PI control. The error in the detected probe signal (e.g., the departure of the split photodetector difference signal from its original reference value) is used as the low voltage input to a high voltage op amp circuit that applies the bias to the piezoelectric positioner to control the probe sample separation distance.

Digital feedback, implemented with the use of digital signal processors to replace hardwired analog components, is used to some degree by most commercial SPM manufacturers. The digital approach has the advantage of providing complete control of the instrument via software, which allows the acquisition of essentially simultaneous images at two different forces. This capability is used for magnetic force imaging, for example, in which the tip of the cantilever is coated with a magnetic material. The topography of the sample is imaged first along each scan line in conventional atomic force mode, then, the probe is lifted to a chosen height above the surface, and scanned across the same scan line, following the exact shape of the topographical information collected during the atomic force scan. Since magnetic (or electrostatic) forces act over longer distances than atomic forces, the second scan detects differences in magnetization.

LIMITATIONS OF SPM INSTRUMENTS

As useful as SPM techniques and instruments may be, they do have limitations that must be considered.

Sample size

The overall dimensions of objects to be measured by SPM are often limited by the requirement that the object be small enough to mount into the instrument. In many AFM's, the object itself is moved in three dimensions by the piezoelectric tube scanner, while the deflections of a stationary cantilever are detected as the probe signal. This restriction stems from the fact that most implementations of AFM use the optical lever arm configuration for detecting cantilever deflections. Moving the cantilever instead of the object would cause the path of the reflected beam to change, altering the detected signal even in the absence of a sample. This limitation has been overcome in several implementations. Digital Instruments' Dimensionª uses a lens mounted in the tube scanner to keep the laser spot focused at the same spot on the cantilever. Park Scientific Instruments has demonstrated an AFM design based on piezoresistive cantilevers, which completely avoids the need for aligned optical components to detect deflections, and thus allows the cantilever to be scanned over the sample.

Scan range

A tradeoff that exists in many measuring devices is that a very sensitive instrument may only be able to measure over a limited range. This is true for the AFM as well. Piezoelectric transducers provide high resolution positioning for SPM probes precisely because they have such a low gain (1 nm to 400 nm per volt). The maximum scan range of a piezoelectric tube is therefore limited to less than 200 µm (0.008") for supply voltages of ± 220 volts.

Piezoelectric transducer nonlinearity

Although piezoelectric transducers allow convenient positioning at or below the nanometer scale, one drawback is that they exhibit hysteretic behavior. That is, position is not a single-valued, linear function of the applied voltage, so, if a certain point on the sample is in the center of the image as the SPM scans in the +x direction, it may not be at the image center when the SPM scans in the -x direction. It is possible to linearize the scan position approximately for most imaging applications by fitting the position vs. bias characteristic to a quadratic equation. However, scanner response also varies with scan frequency, temperature, and even age of the scanner.[4] There is usually also some degree of crosscoupling between signals intended to produce orthogonal motion (e.g., a change in the x position bias can produce a change in the y and/or z position).

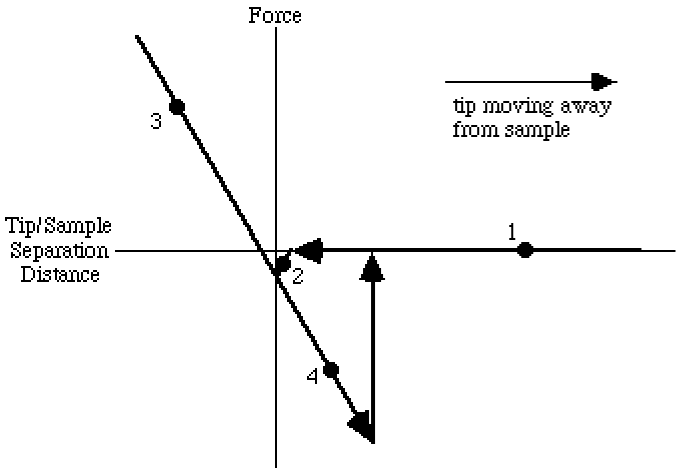
Probe size and shape

Another uncertainty that arises in SPM stems from the finite size and shape of the probe itself, especially when the features to be imaged have high aspect ratios. A probe tip that is large or blunt can measure very flat surfaces without much loss of information, but will not be able to trace the true profile of a surface that includes high aspect ratio features that are sharper, or of smaller dimensions than the probe tip. This is best illustrated by considering the case shown in Figure 6, which shows the effect of probe shape on the measured profile. The tip shape is convoluted with the profile of the surface features, and can dominate a measurement, as well as producing misleading information. Special, high aspect ratio probes with cylindrical shapes, submicron diameters, and lengths of microns or more, have been developed to counter these effects during metrology measurements of x-ray lithography masks, photoresist sidewalls, etc.5, 6 It should be remembered, however, that these problems due to probe dimensions are a reflection of the ultrahigh resolution that SPM can provide.

Figure 6. Effect of probe shape on detected topography.

Problem 3.

Describe the tip-sample interactions at each of the four indicated points on the following cantilever deflection versus separation distance curve. Draw simple sketches of the cantilever deflection (i.e., direction and relative magnitude of cantilever bending) corresponding to each of the 4 points.



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