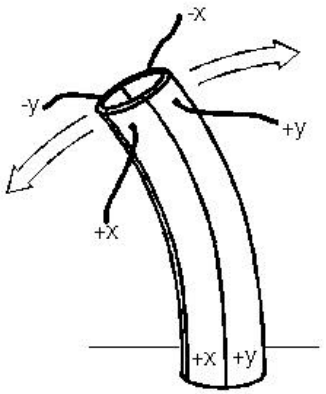
Piezoelectric Scanners

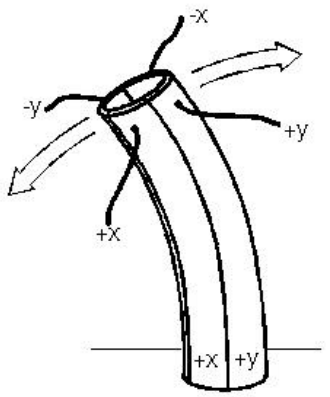
- Piezoelectric materials are ceramics that change dimensions in response to an applied voltage and conversely, they develop an electrical potential in response to mechanical pressure.
- Piezoelectric scanners can be designed to move in x, y, and z by expanding in some directions and contracting in others.
- Usually fabricated from lead zirconium titanate, (PZT) by pressing together a powder, then sintering the material. They are doped to create specific materials properties
- They are polycrystalline solids. Each of the crystals in a piezoelectric material has its own electric dipole moment. These dipole moments are the basis of the scanner's ability to move in response to an applied voltage.



- After sintering, the dipole moments within the scanner are randomly aligned.
- If the dipole moments are not aligned, the scanner has almost no ability to move. A process called poling is used to align the dipole moments.
- During poling the scanners are heated to about 200°C to free the dipoles, and a DC voltage is applied to the scanner. Within hours most of the dipoles become aligned. At that point, the scanner is cooled to freeze the dipoles into their aligned state.
- Occasional use of the scanner maintains the scanner's polarization. The voltage applied to enact the scanning motion realigns the stray dipoles that relax into random orientation. If the scanner is not re-poled by regular use, a significant fraction of the dipoles will begin to randomize (depolarize or de-pole) again over a period of weeks (or months).
- De-poling is accelerated markedly if the scanner is subjected to temperatures above 150°C. This means that if you want to add a heated stage to your SPM, you must isolate it thermally from the scanner (The Curie temperature for PZT materials is about 150°C.)

Piezoelectric Scanners

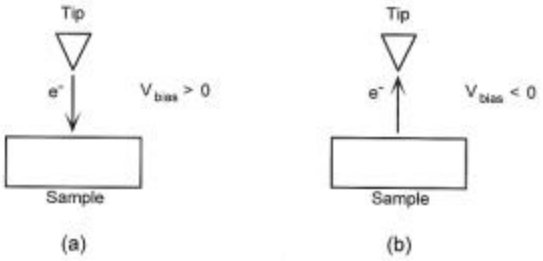
- **Electrodes are attached to the outside of the tube, segmenting it electrically into vertical quarters, for +x, +y, -x, and -y travel. An electrode is also attached to the center of the tube to provide motion in the z direction.**
- **When alternating voltages are applied to the +x and -x electrodes, the induced strain of the tube causes it to bend back and forth in the x direction. Voltages applied to the z electrode cause the scanner to extend or contract vertically.**
- **In most cases, the voltage applied to the z electrode of the scanner at each measurement point constitutes the AFM (constant-force) or the STM (constant-current) data set.**
- **An external sensor can be used to measure the height of the scanner directly (Hardware Correction).**

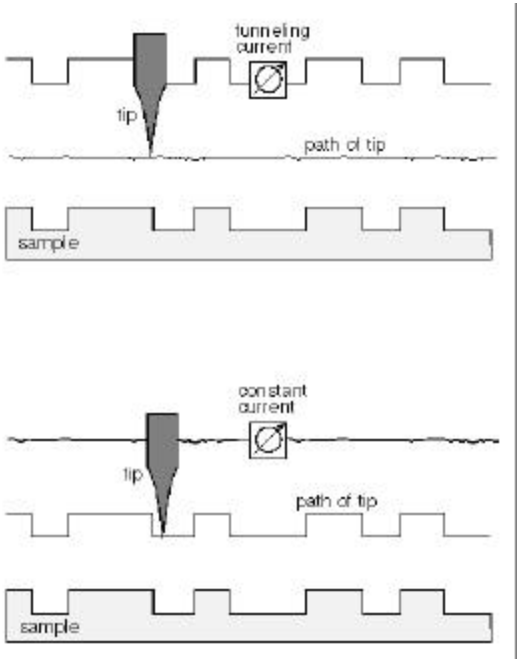


- **The maximum scan size that can be achieved with a particular piezoelectric scanner depends upon the length of the scanner tube, the diameter of the tube, its wall thickness, and the strain coefficients of the particular piezoelectric ceramic from which it is fabricated.**
- **Typical SPMs scanners can scan laterally from tens of angstroms to over 100 microns. In the vertical direction, SPM scanners can distinguish height variations from the sub-angstrom range to about 10 microns.**
- **Piezoelectric scanners are critical elements in SPMs, valued for their sub-angstrom resolution, their compactness, and their high-speed response. However, along with these essential properties come some challenges due to scanner nonlinearities.**

Scanning Tunneling Microscopy (STM)

- Each mode has advantages and disadvantages. Constant-height mode is faster because the system doesn't have to move the scanner up and down, but it provides useful information only for relatively smooth surfaces. Constant-current mode can measure irregular surfaces with high precision, but the measurement takes more time.
- As a first approximation, an image of the tunneling current maps the topography of the sample. More accurately, the tunneling current corresponds to the electronic density of states at the surface. STMs actually sense the number of filled or unfilled electron states near the Fermi surface, within an energy range determined by the bias voltage. Rather than measuring physical topography, it measures a surface of constant tunneling probability.
- The sensitivity of STMs to local electronic structure can cause trouble if interested in mapping topography. For example, if an area of the sample has oxidized, the tunneling current will drop precipitously when the tip encounters that area. In constant-current mode, the STM will instruct the tip to move closer to maintain the set tunneling current. The result may be that the tip digs a hole in the surface.
- The sensitivity of STMs to electronic structure can, however, be a tremendous advantage. Other techniques for obtaining information about the electronic properties of a sample detect and average the data originating from a relatively large area, a few microns to a few millimeters across. STMs can be used as surface analysis tools that probe the electronic properties of the sample surface with atomic resolution.





Bias Voltage in STM

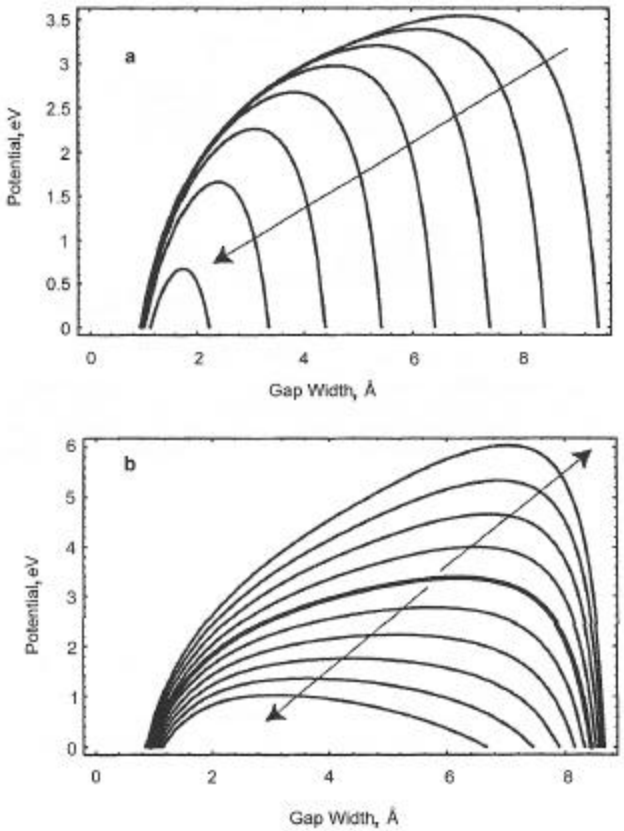
STM Tips:

Tungsten, gold, Pt/Ir (not oxidized), Rh/Ir

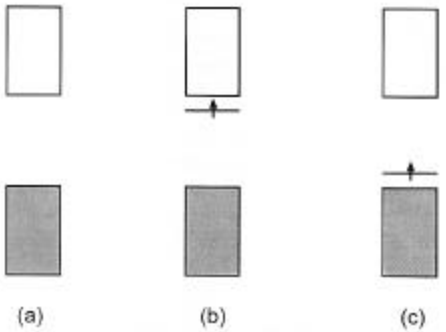
Fabricated by mechanical cutting, electrochemical etching

Tunneling current: 0.1-40 nA

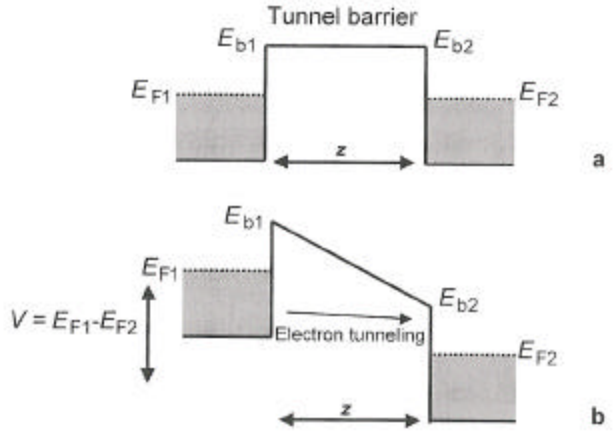
Bias voltage: 10^{-3} – 5 V



A more realistic depiction of the tunneling barrier, including the effect of image charge on the shape of the barrier. **a**, As the distance between the solids is decreased (*arrow*), the size and shape of the barrier change. **b**, The barrier also changes when a voltage is applied, the arrows show the response to opposite signs of bias.[20]



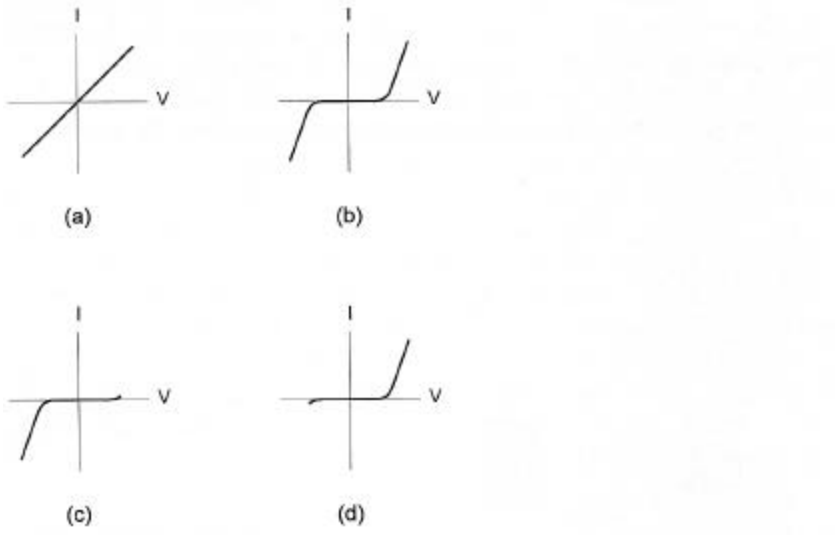
Energy levels of regular semiconductors in the vicinity of their band gaps: (a) de fect-free semiconductor; (b) n-type semiconductor; (c) p-type semiconductor.



The energy levels in two solids separated by an insulating or vacuum barrier (**a**) with no bias applied between the solids and (**b**) with an applied bias. Energies of the electrons in the solids are indicated by the shaded areas up to E_{F1} and E_{F2}, which are the Fermi levels of the respective materials. The applied bias V is $E_{F1}-E_{F2}$, and z is the distance between the two solids.

Scanning Tunneling Spectroscopy

- **Scanning tunneling spectroscopy (STS) studies the local electronic structure of a sample's surface.**
- **The electronic structure of an atom depends upon its atomic species and upon its local chemical environment (how many neighbors it has, what kind of atoms they are, and the symmetry of their distribution).**



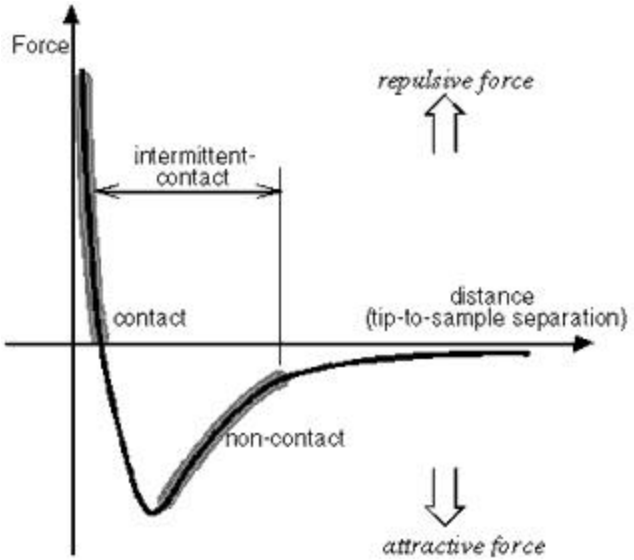
Schematic representations of the $I-V$ curves expected for metals and regular semiconductors: (a) metals; (b) defect-free semiconductors; (c) n-type semiconductors; (d) p-type semiconductors.

STS encompasses many methods:

- **Taking "topographic" (constant-current) images using different bias voltages and comparing them; taking current (constant-height) images at different heights; and ramping the bias voltage with the tip positioned over a feature of interest while recording the tunneling current.**
- **The last example results in current vs. voltage (I-V) curves characteristic of the electronic structure at a specific x,y location on the sample surface.**
- **STMs can be set up to collect I-V curves at every point in a data set, providing a three-dimensional map of electronic structure. With a lock-in amplifier, dI/dV (conductivity) or dI/dz (work function) vs. V curves can be collected directly.**
- **All of these are ways of probing the local electronic structure of a surface using an STM.**

Contact Mode AFM

- **The atomic force microscope (AFM) probes the surface of a sample with a couple of microns long sharp tip, often less than 100Å in diameter. The tip is located at the free end of a cantilever that is 100 to 200μm long.**
- **Forces between the tip and the sample surface cause the cantilever to bend, or deflect. A detector measures the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under to generate a map of surface topography.**
- **AFMs can be used to study insulators and semiconductors as well as electrical conductors.**
- **Several forces typically contribute to the deflection of an AFM cantilever. The force most commonly associated with atomic force microscopy is an interatomic force called the van der Waals force.**



Interatomic force vs. distance curve

Two distance regimes:

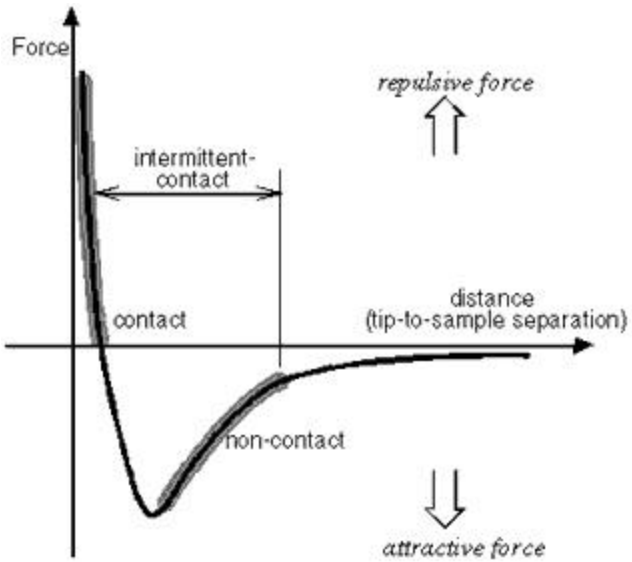
1) the contact regime; and 2) the non-contact regime.

In the contact regime, the cantilever is held less than a few angstroms from the sample surface, and the interatomic force between the cantilever and the sample is repulsive.

In the non-contact regime, the cantilever is held on the order of tens to hundreds of angstroms from the sample surface, and the interatomic force between the cantilever and sample is attractive (largely a result of the long-range van der Waals interactions).

Contact Mode AFM

- In contact AFM mode, also known as repulsive mode, an AFM tip makes soft "physical contact" with the sample. The tip is attached to the end of a cantilever with a low spring constant, lower than the effective spring constant holding the atoms of the sample together. As the scanner gently traces the tip across the sample the contact force causes the cantilever to bend to accommodate changes in topography.



Interatomic force vs. distance curve

- As the atoms are gradually brought together, they first weakly attract each other. This attraction increases until the atoms are so close together that their electron clouds begin to repel each other electrostatically. This electrostatic repulsion progressively weakens the attractive force as the interatomic separation continues to decrease. The force goes to zero when the distance between the atoms reaches a couple of angstroms, about the length of a chemical bond. When the total van der Waals force becomes positive (repulsive), the atoms are in contact.

- The slope of the van der Waals curve is very steep in the repulsive or contact regime. As a result, the repulsive van der Waals force balances almost any force that attempts to push the atoms closer together. In AFM this means that when the cantilever pushes the tip against the sample, the cantilever bends rather than forcing the tip atoms closer to the sample atoms. Even if you design a very stiff cantilever to exert large forces on the sample, the interatomic separation between the tip and sample atoms is unlikely to decrease much.

Contact Mode AFM

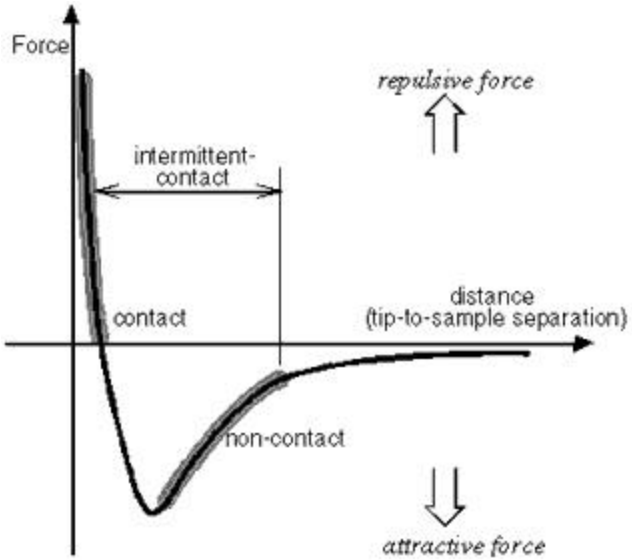
- **Two other forces are generally present during contact AFM operation: a capillary force exerted by the thin water layer often present in an ambient environment, and the force exerted by the cantilever itself.**
- **The capillary force arises when water surrounds the tip, applying a strong attractive force (about 10^{-8} N) that holds the tip in contact with the surface. The magnitude of the capillary force depends upon the tip-to-sample separation. The force exerted by the cantilever is like the force of a compressed spring. The magnitude and sign (repulsive or attractive) of the cantilever force depends upon the deflection of the cantilever and upon its spring constant.**
- **As long as the tip is in contact with the sample, the capillary force should be constant because the distance between the tip and the sample is virtually incompressible.**
- **It is assumed that the water layer is reasonably homogeneous. The variable force in contact AFM is the force exerted by the cantilever. The total force that the tip exerts on the sample is the sum of the capillary plus cantilever forces, and must be balanced by the repulsive van der Waals force for contact AFM. The magnitude of the total force exerted on the sample varies from 10^{-8} (with the cantilever pulling away from the sample almost as hard as the water is pulling down the tip), to the more typical operating range of 10^{-7} to 10^{-6} N.**
- **Most AFMs detect the position of the cantilever with optical techniques. In the most common scheme, a laser beam bounces off the back of the cantilever onto a position-sensitive photodetector (PSPD). As the cantilever bends, the position of the laser beam on the detector shifts.**
- **The PSPD itself can measure displacements of light as small as 10Å. The ratio of the path length between the cantilever and the detector to the length of the cantilever itself produces a mechanical amplification. As a result, the system can detect sub-angstrom vertical movement of the cantilever tip.**

Contact Mode AFM

- Other methods of detecting cantilever deflection rely on optical interference, or even a scanning tunneling microscope tip to read the cantilever deflection. One particularly elegant technique is to fabricate the cantilever from a piezoresistive material so that its deflection can be detected electrically. (In piezoresistive materials, strain from mechanical deformation causes a change in the material's resistivity.) For piezoresistive detection, a laser beam and a PSPD are not necessary.
- Once the AFM has detected the cantilever deflection, it can generate the topographic data set by operating in one of two modes:
- In constant-height mode, the spatial variation of the cantilever deflection can be used directly to generate the topographic data set because the height of the scanner is fixed as it scans.
- In constant-force mode, the deflection of the cantilever can be used as input to a feedback circuit that moves the scanner up and down in z, responding to the topography by keeping the cantilever deflection constant. In this case, the image is generated from the scanner's motion. With the cantilever deflection held constant, the total force applied to the sample is constant.
- In constant-force mode, the speed of scanning is limited by the response time of the feedback circuit, but the total force exerted on the sample by the tip is well controlled. Constant-force mode is generally preferred for most applications.
- Constant-height mode is often used for taking atomic-scale images of atomically flat surfaces, where the cantilever deflections and thus variations in applied force are small. Constant-height mode is also the way to record real-time images of changing surfaces, where high scan speed is essential.

Non-Contact Mode AFM

- Non-contact AFM (NC-AFM) an AFM cantilever is vibrated near the surface of a sample. The spacing between the tip and the sample for NC-AFM is on the order of tens to hundreds of angstroms. NC-AFM is desirable because it provides a means for measuring sample topography with little or no contact between the tip and the sample.
- Like contact AFM, non-contact AFM can be used to measure the topography of insulators and semiconductors as well as electrical conductors. The total force between the tip and the sample in the non-contact regime is very low, generally about 10^{-12} N. This low force is advantageous for studying soft or elastic samples.
- Sensitive samples, like silicon wafers, are not contaminated through contact with the tip.

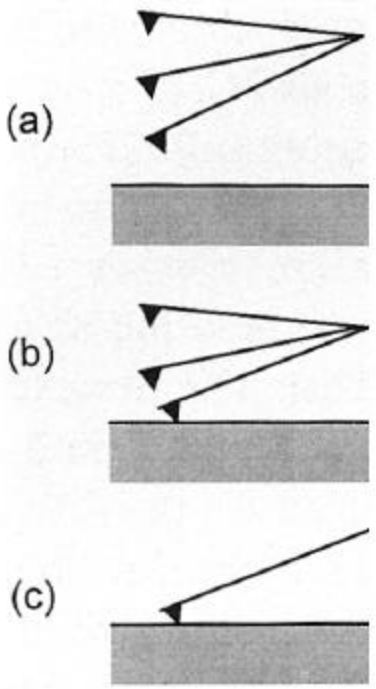


Interatomic force vs. distance curve

- Because the force between the tip and the sample in the non-contact regime is low, it is more difficult to measure than the force in the contact regime, which can be several orders of magnitude greater.
- Cantilevers used for NC-AFM must be stiffer than those used for contact AFM because soft cantilevers can be pulled into contact with the sample surface. The small force values in the non-contact regime and the greater stiffness of the cantilevers used for NC-AFM are both factors that make the NC-AFM signal small, and therefore difficult to measure. Thus, a sensitive, AC detection scheme is used for NC-AFM operation.
- In non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically from 100 to 400 kHz) with an amplitude of a few tens of angstroms. Then it detects changes in the resonant frequency or vibration amplitude as the tip comes near the sample surface. The sensitivity of this detection scheme provides sub-angstrom vertical resolution in the image, as with contact AFM.

Non-Contact Mode AFM

- The relationship between the resonant frequency of the cantilever and variations in sample topography can be explained as follows:
- The resonant frequency of a cantilever varies as the square root of its spring constant. In addition, the spring constant of the cantilever varies with the force gradient experienced by the cantilever.
- Finally, the force gradient, which is the derivative of the force versus distance curve changes with tip-to-sample separation. Thus, changes in the resonant frequency of a cantilever can be used as a measure of changes in the force gradient, which reflect changes in the tip-to-sample spacing, or sample topography.
- In NC-AFM mode, the system monitors the resonant frequency or vibrational amplitude of the cantilever and keeps it constant with the aid of a feedback system that moves the scanner up and down. By keeping the resonant frequency or amplitude constant, the system also keeps the average tip-to-sample distance constant.
- As with contact AFM (in constant-force mode), the motion of the scanner is used to generate the data set.
- NC-AFM does not suffer from the tip or sample degradation effects that are observed after taking several scans with contact AFM.
- NC-AFM is also preferable to contact AFM for measuring soft samples. In the case of rigid samples, contact and non-contact images may look the same.



(a) noncontact mode;
(b) tapping mode;
(c) force-modulation mode.

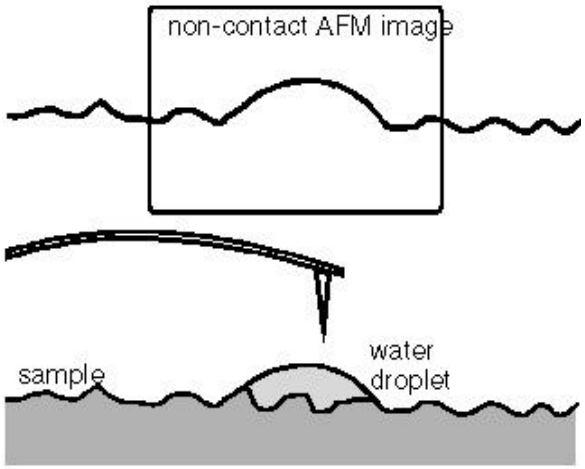
Resonant frequency of an AFM tip:

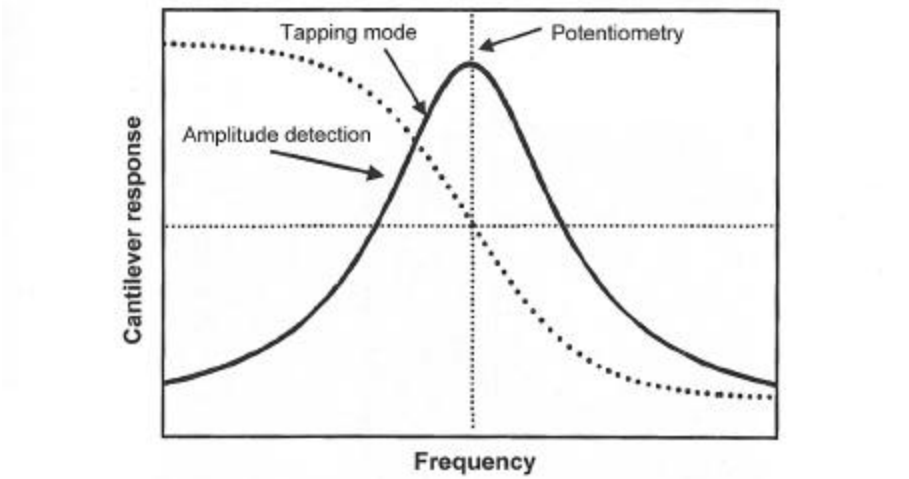
$$w \propto \frac{t}{l^2}\sqrt{\frac{E}{r}} = \sqrt{\frac{k}{m_{eff}}}$$

Non-contact *vs.* contact AFM

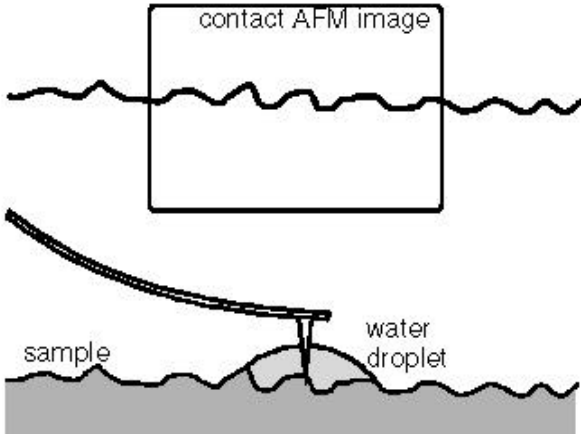
- **If a few monolayers of condensed water are lying on the surface of a rigid sample, the image may look quite different. An AFM operating in contact mode will penetrate the liquid layer to image the underlying surface, whereas in non-contact mode an AFM will image the surface of the liquid layer.**

- **For cases where a sample of low moduli may be damaged by the dragging of an AFM tip across its surface, intermittent-contact mode of AFM is available.**

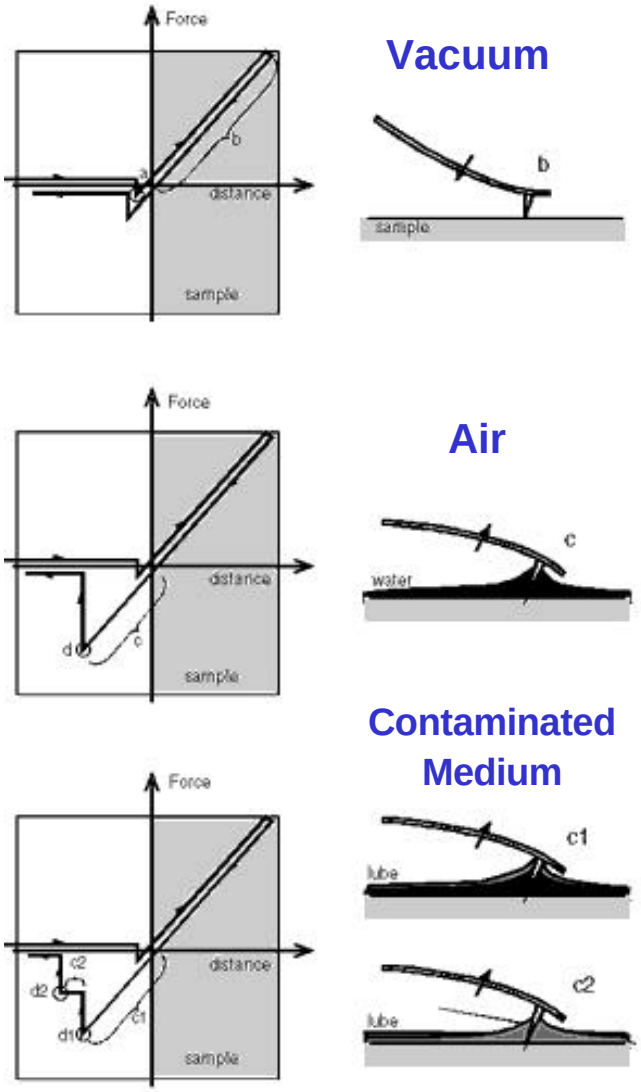




Frequency response of a typical AFM cantilever. The amplitude of the tip vibration as a function of driving frequency exhibits a peak that indicates the tip resonant frequency. The second line shows the phase of the cantilever oscillation.

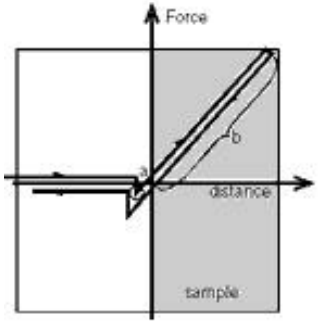


Force-Distance Curves

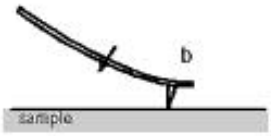


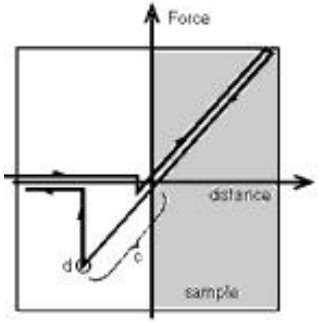
- Force vs. distance curves are used to measure the vertical force that the tip applies to the surface while a contact-AFM image is being taken. This technique can also be used to analyze surface contaminants' viscosity, lubrication thickness, and local variations in the elastic properties of the surface.
- A force vs. distance curve is a plot of the deflection of the cantilever versus the extension of the piezoelectric scanner, measured using a position-sensitive photodetector. The van der Waals force curve represents just one contribution to the cantilever deflection. Local variations in the form of the F vs. d curve indicate variations in the local elastic properties. Contaminants and lubricants affect the measurement, as does the thin layer of water present when operating an AFM in air.
- Consider the simplest case of AFM in vacuum. At the left side of the curve, the scanner is fully retracted and the cantilever is undeflected since the tip is not touching the sample. As the scanner extends, the cantilever remains undeflected until it comes close enough to the sample surface for the tip to experience the attractive van der Waals force. The tip snaps into the surface (point a). Equivalently, the cantilever suddenly bends slightly towards the surface.
- As the scanner continues to extend, the cantilever deflects away from the surface, approximately linearly (region b). After full extension, at the extreme right of the plot, the scanner begins to retract. The cantilever deflection retraces the same curve (in the absence of scanner hysteresis) as the scanner pulls the tip away from the surface.

Force-Distance Curves



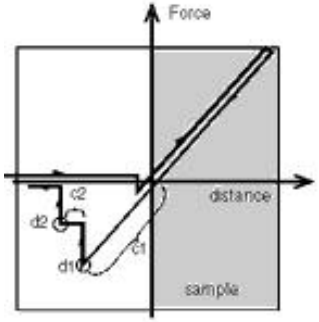
Vacuum

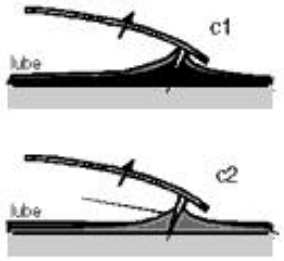




Air

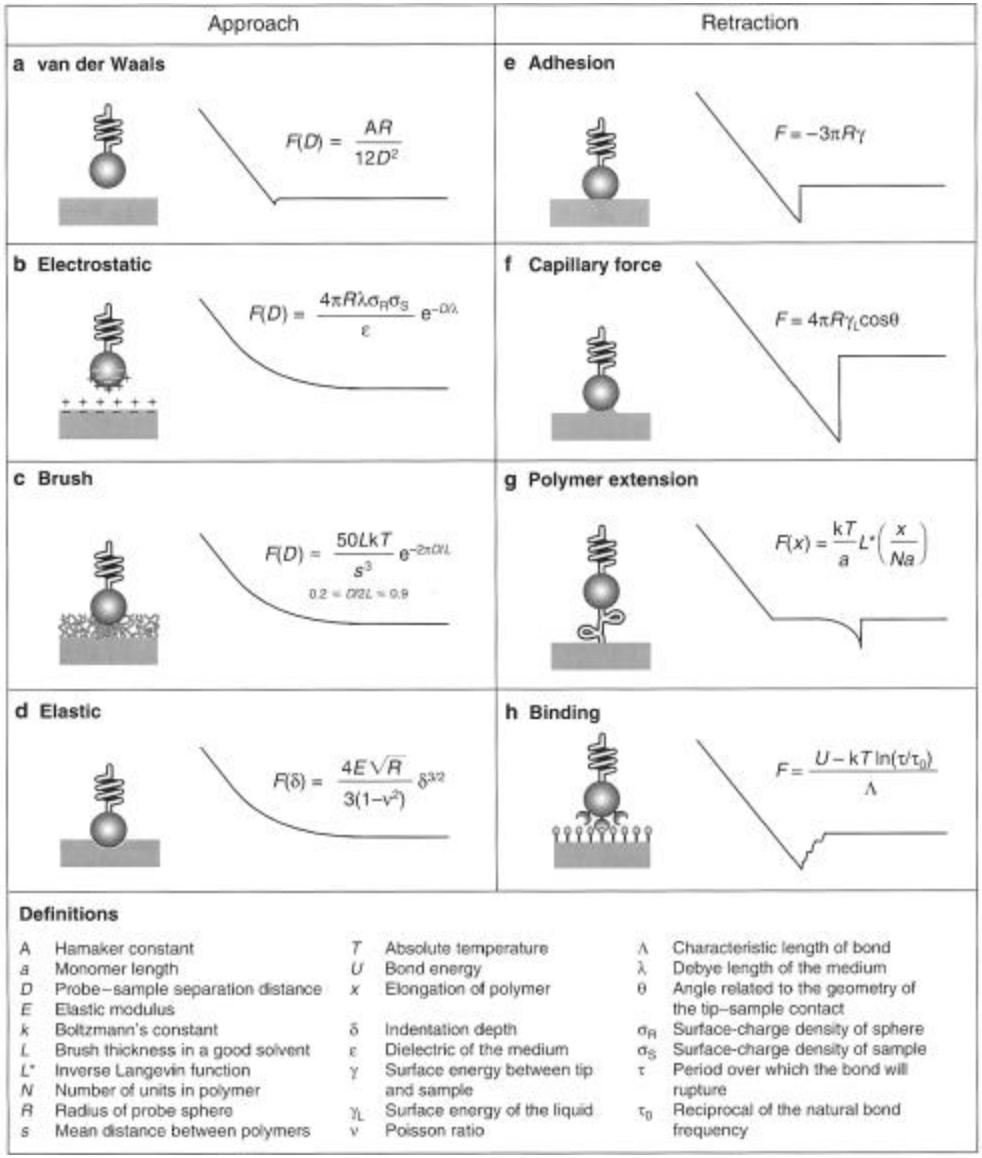
Contaminated Medium





- In air, **the retracting curve is often different because** a monolayer or a few monolayers of water are present on many surfaces. **This water layer exerts a capillary force that is very strong and attractive. As the scanner pulls away from the surface, the water holds the tip in contact with the surface, bending the cantilever strongly towards the surface (region c). At some point, depending upon the thickness of the water layer, the scanner retracts enough that the tip springs free (point d). This is known as the snap-back point.**
- If a lubrication layer is present along with the water layer, multiple snap-back points can occur. **The positions and amplitudes of the snap-back points depend upon the viscosity and thickness of the layers present on the surface.**
- Contact AFM can be operated anywhere along the linear portion of the force vs. distance curves, in regions (b) or (c). **Operation in region (c) might be used for soft samples to minimize the total force on the sample. Operating with the cantilever bent towards the surface is inherently a less stable situation, and maximum scan speeds may have to be reduced. Operation in region (c) is still called contact mode, since the tip is touching the sample.** Non-contact AFM is operated just to the left of point (a) on the F vs. d curve-just before the tip snaps into the surface.
- In the linear region of the F vs. d curves (b), the slope is related to the elastic modulus of the system. **When the cantilever is much softer than the sample surface, the slope of the curve mostly reflects the spring constant of the cantilever. When the cantilever is much stiffer than the sample, the slope of the F vs. d curve allows investigation of the elastic properties of the sample.**

Force-Distance Curves



a. Ideal, attractive, van der Waals force in the absence of other forces

b. Repulsive electrostatic double-layer force in solution

c. Polymer-brushing forces that result from the thermally driven motion of polymers grafted onto a solid surface in solution

d. Indentation curve on an elastic sample; In this case, the true contact is near the point at which the cantilever begins to deflect

e. Adhesion between a sphere and a plane in the absence of contaminating adsorbates (typically in a vacuum)

f. Capillary adhesion (very common under ambient conditions, under which many surfaces have thin layers of water) results from the formation of a water bridge between the tip and sample

g. Polymer-extension force curves with a negative deflection far from the surface and a jump back to zero deflection as the polymer breaks or detaches from one of the surfaces

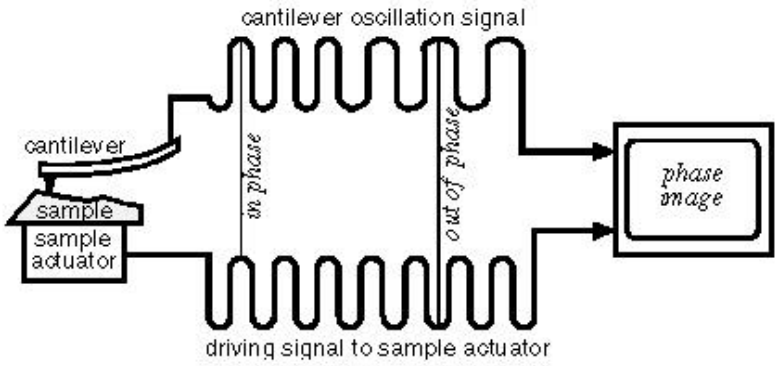
h. The unbinding of receptor–ligand pairs produces stepwise return to zero deflection from the point of maximal adhesion. This is due to sequential unbinding of multiple receptor–ligand pairs.

Phase imaging (Phase Detection Microscopy)

Phase Detection Microscopy (PDM) (or phase imaging) is used to map variations in surface properties such as elasticity, adhesion, and friction.

Phase detection images can be produced while an instrument is operating in any vibrating cantilever mode, such as NC-AFM, intermittent-contact AFM, MFM and FMM mode.

The phase lag between the signal that drives the cantilever to oscillate and the cantilever oscillation output signal is monitored. Changes in the phase lag reflect changes in the mechanical properties of the sample surface.



The system feedback loop uses changes in the cantilever's deflection to measure sample topography. The phase lag is monitored while the topographic image is being taken so that images of topography and material properties are collected simultaneously.

Phase detection can be used to obtain material-properties for samples whose topography is best measured using non-contact rather than contact AFM.

Phase detection is an alternative to Force Modulation AFM, which uses contact AFM.

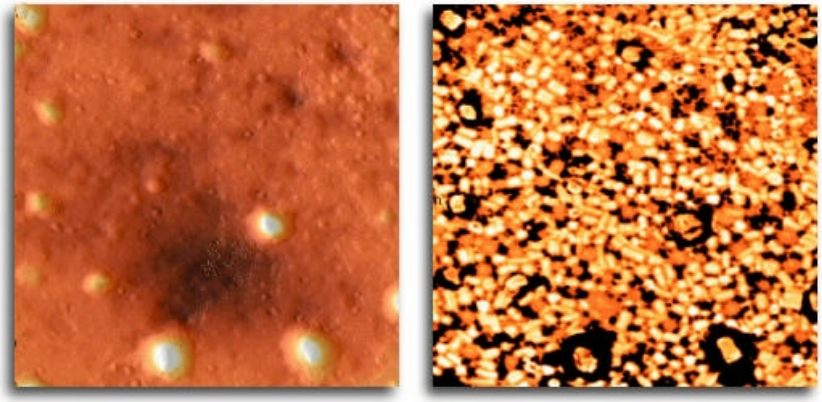
Applications

Polymers, blends, fiber coatings, metal coatings

Thin film coatings, lubrication films, defects, grain boundaries

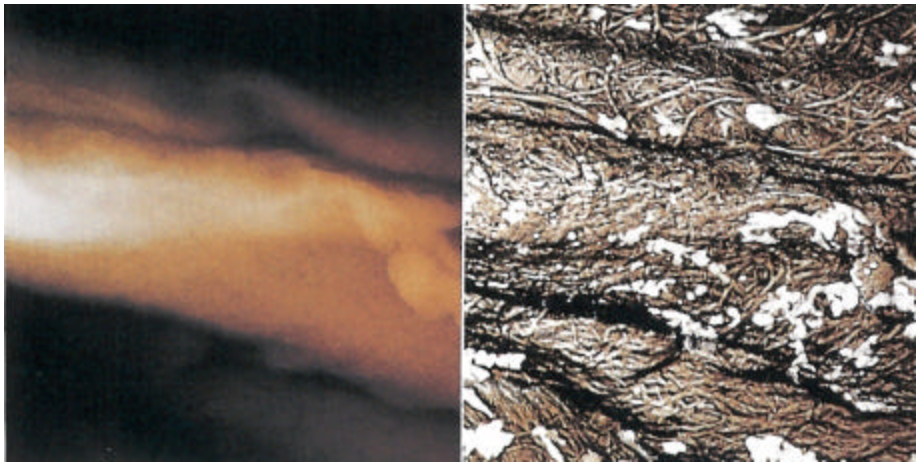
Soft biological samples and macro-molecules

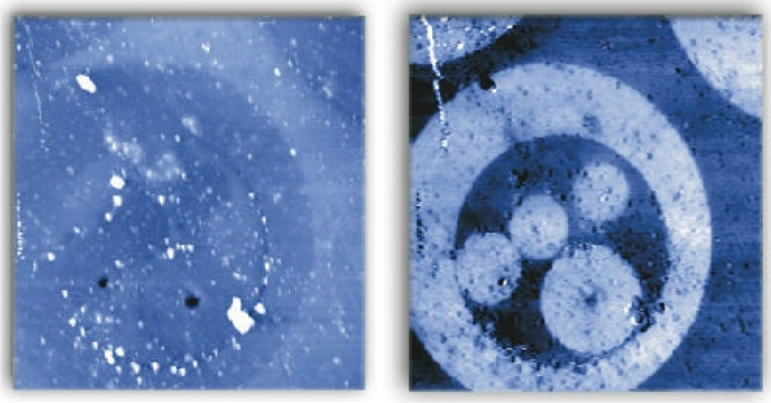
Phase Images



Field of view 5µm x 5µm

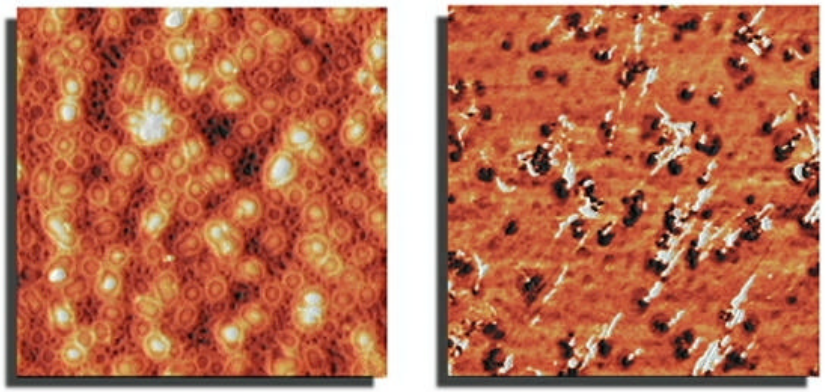
Teflon particles in a Fluoropolymer matrix are seen in the Topography image (left) and the Phase Signal image (right). The particulate nature of the Fluoropolymer matrix is seen clearly in the Phase Signal image.





Field of view 50µm x 50µm

The image on the left is a topographical image taken in Contact-AFM. Simultaniously, the image on the right corresponds to the local visco-elasticity properties taken with the Phase signal. In this example, the phase signal is sensitive to the various phases of the polymer: the matrix is high density polyethylen and the disordered phase is polystyren.



Field of view 9µm x 9µm

Topography image (left) of teflon particles in support coating. Phase imaging (right) of teflon particles in support coating. Note the clear location of the teflon particles in both topography (bright area)and phase images (dark areas). The trace silicon lube (white areas) can also be observed in the phase image.

Force Modulation Microscopy (FMM)

Force modulation microscopy (FMM) is an extension of contact AFM. It allows for simultaneous acquisition of both topographic and material properties data.

The AFM tip is in contact with the sample, and the z feedback loop maintains a constant cantilever deflection (constant-force mode AFM).

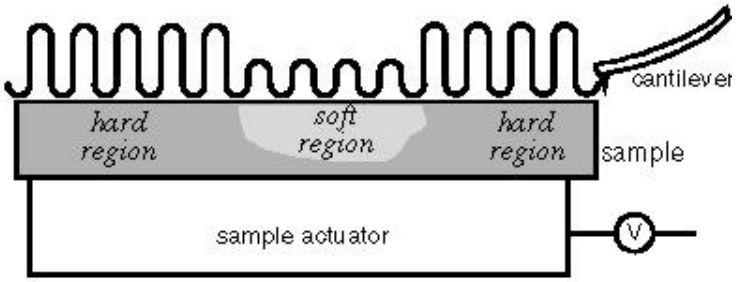
A periodic signal is applied to either the tip or the sample. The amplitude of cantilever modulation that results from this applied signal varies according to the elastic properties of the sample.

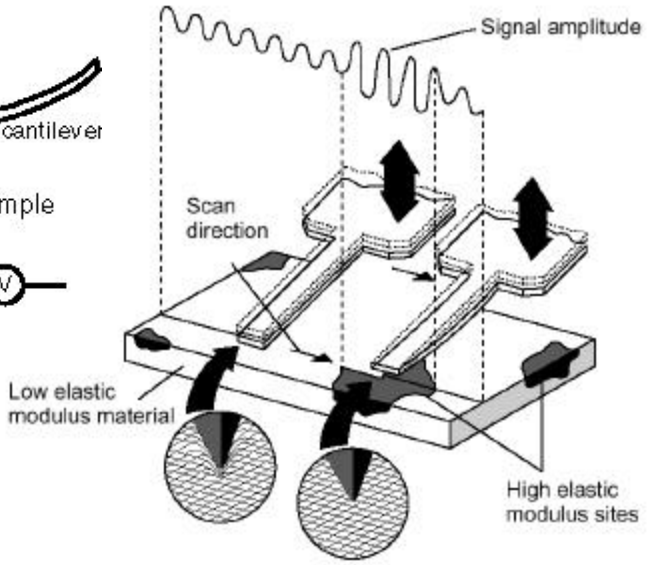
The force modulation image, which is a map of the elastic properties of the sample, from the changes in the amplitude of the cantilever modulation.

The frequency of the applied signal is on the order of hundreds of kilohertz, which is faster than the z feedback loop. Thus, topographic information can be separated from local variations of the sample elastic properties, and the two types of images are collected simultaneously.

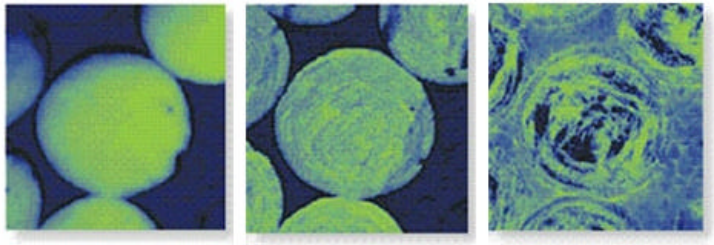
Applications

- composites
- blended polymers
- structural materials
- rubbers and plastics
- adhesives
- coatings
- surface contaminants
- subsurface structures



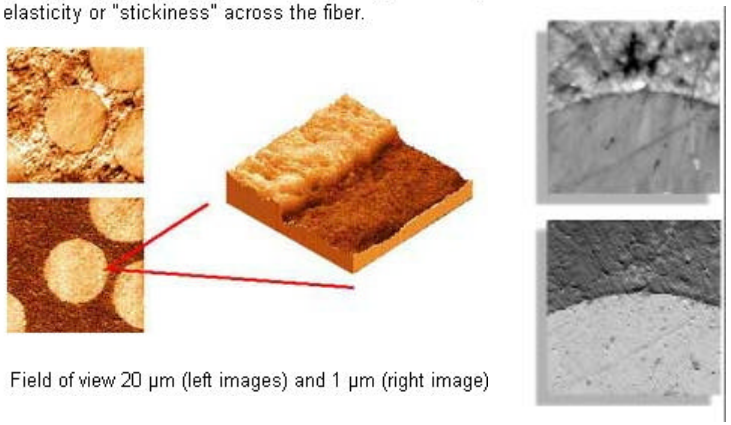


Force Modulation Images



Field of view 15 µm x 15 µm

Contact AFM images of carbon fiber-epoxy composite acquired simultaneously in topography (left), force modulation (center), and phase detection (right) modes. The force modulation image shows the harder (brighter) carbon fiber in the darker colored epoxy matrix. The concentric rings visible within the fiber are probably caused by the diffusion of oxygen through the fiber during the transformation of the fiber from a polymeric to a graphite state. The phase image shows similar image contrast, and shows differences in the elasticity or "stickiness" across the fiber.

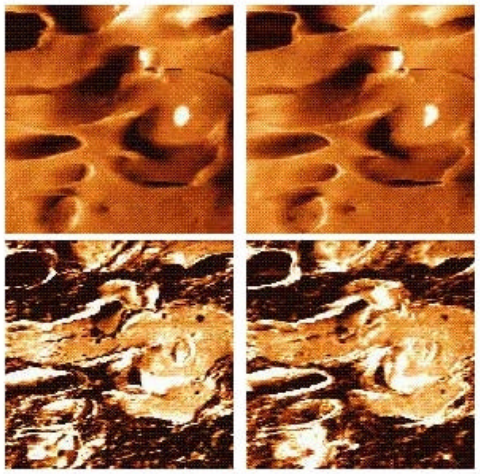


Field of view 20 µm (left images) and 1 µm (right image)

Topographic contact-AFM image (top left) and dF/dz force modulation image (bottom left) of a fiber/polymer composite, measured simultaneously. The force modulation data clearly show the contrast between the harder fiber (bright areas) and the softer polymer (dark areas). The three dimensional rendition of the force modulation image (right) with higher resolution shows the variation in the fiber/polymer interface properties.

Force modulation obtains high resolution elasticity information by vibrating either the tip or the sample in contact AFM mode and measuring the corresponding deflection of the AFM cantilever, in effect measuring the local slope of the force vs. distance curve. Amplitude measurements show variations in the local elasticity and hardness of the sample in the tip region.

This elastic information is substantially decoupled from topographic variations and permits identification and differentiation of materials by their physical properties. The extremely high lateral spatial resolution 10 nm order allows the transition interface between the materials to be studied in detail.

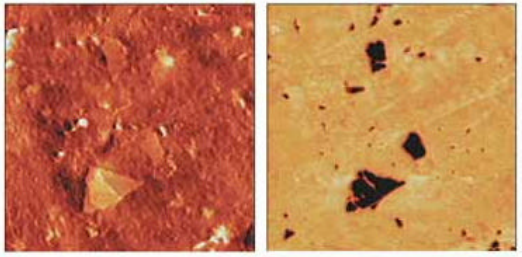


Field of view 20 µm x 20 µm

Simultaneously recorded topography (top) and dF/dz images (bottom) of cross-sectioned blended polymer (bright hard, dark soft). Images are recorded in both scan directions to check for artifacts.

Force modulation obtains high resolution elasticity information by vibrating the tip or sample in contact AFM mode and measuring the corresponding deflection of the AFM cantilever, in effect measuring the local slope of the force vs. distance curve, dF/dz. Amplitude measurements show variations in the local elasticity and hardness of the sample in the tip region, whilst phase measurements reveal local viscosity variations.

This elastic information is substantially decoupled from topographic variations and permits identification and differentiation of materials by their physical properties. Quantitative elasticity information can be calculated provided the tip shape and cantilever force constant are known. Accurate z calibration is essential for this application, and ScanMaster z sensors can be used to great advantage.



Field of View 2.95µm x 2.95µm

Topography image (left) and force modulation image (right) of a plastic in plastic polymer blend used to manufacture golfballs.

Lateral Force Microscopy (LFM)

Lateral force microscopy (LFM) measures lateral deflections (twisting) of the cantilever that arise from forces on the cantilever parallel to the plane of the sample surface.

LFM can image variations in surface friction and enhance the edge of any surface image.

Lateral deflections of the cantilever usually arise from two sources: changes in surface friction (roughness) and changes in slope.

(a) the tip may experience greater friction as it traverses some areas, causing the cantilever to twist strongly.

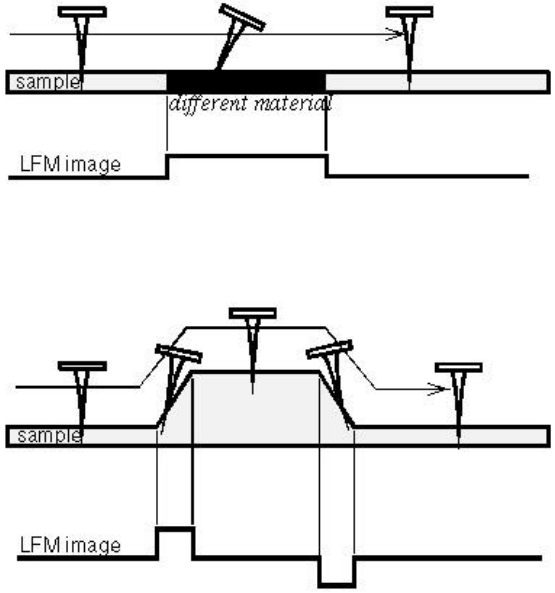
(b) the cantilever may twist when it encounters a steep slope. To separate one effect from the other, LFM and AFM images should be collected simultaneously.

LFM uses a photodetector (PSPD) to detect the deflection of the cantilever. The difference for LFM is that the PSPD also senses the cantilever's twist, or lateral deflection.

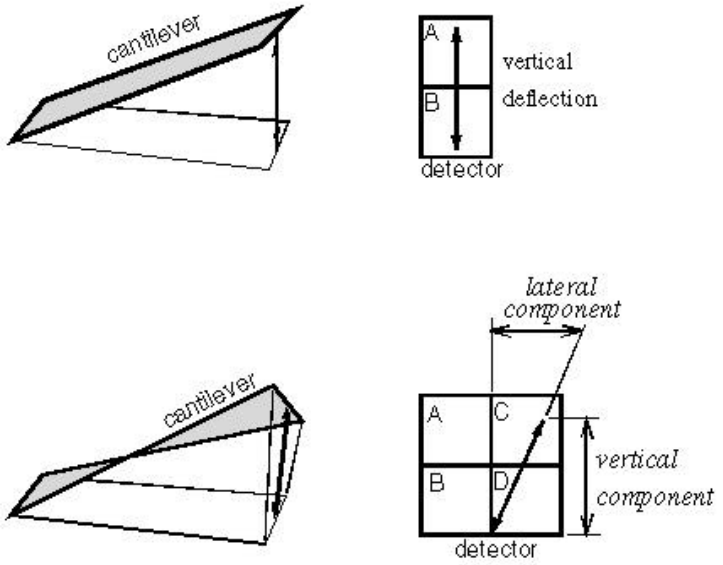
AFM uses a "bi-cell" PSPD, divided into A and B.

LFM requires a "quad-cell" PSPD, divided into four quadrants, A through D. By adding the signals from the A and C quadrants, and comparing the result to the sum from the B and D quadrants, the quad-cell can sense the lateral component of the cantilever's deflection.

AFM and LFM data can be generated simultaneously.

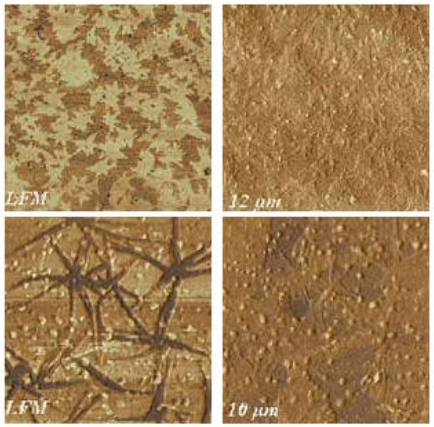


Lateral deflection of the cantilever from changes in surface friction (top) and from changes in slope (bottom).

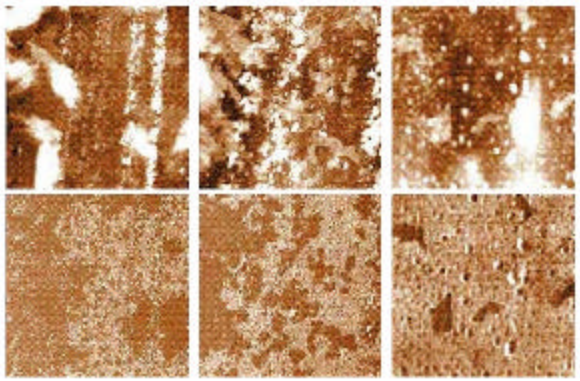


The PSPD for AFM (top) and LFM (bottom)

LFM Images



A metal layer was deposited onto a glass surface. Lateral Force Microscopy (LFM) reveals differences in friction betwee the metal and the glass substrate. Different deposition processes lead to different appeareance of the metallic areas.

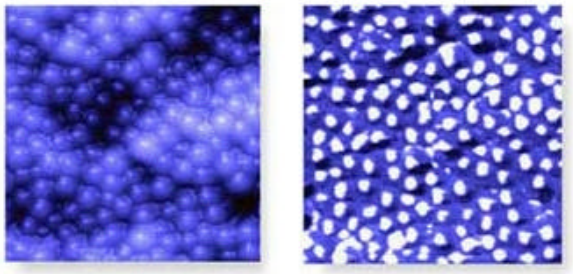


Field of view 1.8 µm x 1.8 µm

Topographic (top) and LFM (bottom) images of Polyacetate surface, scanned left-to-right. High lateral force is bright, low force is dark. Image pairs recorded simultaneously.

Lateral Force Microscopy (LFM) images provide a simple means of discriminating between different materials and calculating the area fraction of each. Low molecular weight monomers integrate poorly and form blobs on the surface; these are seen as low friction (dark) regions in the LFM data. LFM measures torsional deflections of the cantilever induced by lateral forces acting on the tip during scanning. Both frictional and topographic variations contribute to the rotation of the cantilever. ProScan multi-channel acquisition software is used to record topographic AFM and LFM images simultaneously. Precise frictional coefficient determination requires characterization of tip geometry and cantilever stiffness.

Forward (left) and reverse (right) direction LFM images of thiolipid on mica.Thiolipid forms a self-as-sembled monolayer that can protect surfaces from corrosion yet a simple chemical process easily re-moves the layer. These properties make thiolipid an ideal coating for lithographic processes where it can shield underlying substrate material from etching. The flower-shaped condensed domain can be seen against the disordered phase. The internal structure of the domain is revealed by high friction contrast between the different petals.



Field of view 15 µm x 15 µm

Simultaneous C-AFM images of composite latex spheres, composed of a hard core surrounded by a soft shell, showing topography (left) and force modulation (right) image contrast. The force modulation image shows the harder (brighter) core surrounded by the softer (darker) core. Although visible by other microscopy techniques, the two layers of the spheres are visible without any special sample preparation, such as heavy metal staining or metal coating.

Magnetic Force Microscopy (MFM)

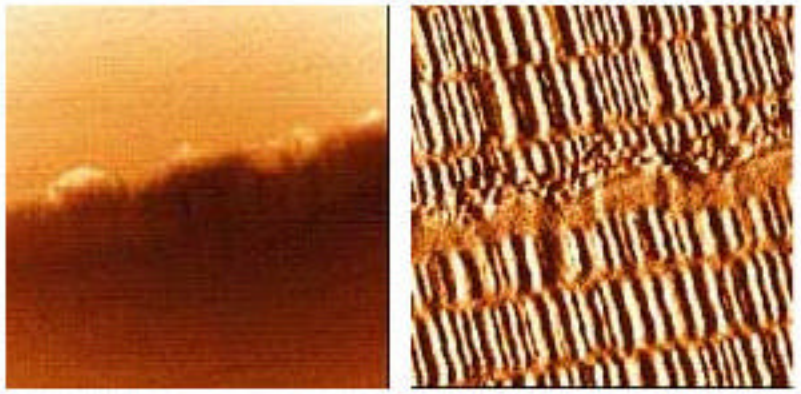
Applications

Media for data storage , hard disks, magnetic films

Signals Measured to Generate Data

MFM Amplitude

MFM Phase



Effects of a head/disk crash. Topography (left) and Magnetic Force Microscopy (right).

The system operates in non-contact, detecting changes in the resonant frequency of the cantilever due to the dependence of the magnetic field on tip-to-sample separation.

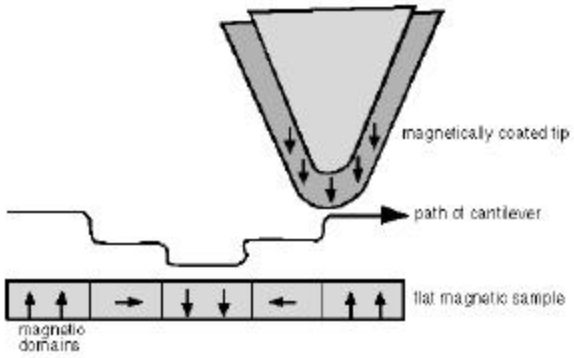
MFM is used to image domain structures in magnetic materials.

An MFM image contains information about both the topography and the magnetic properties of a surface.

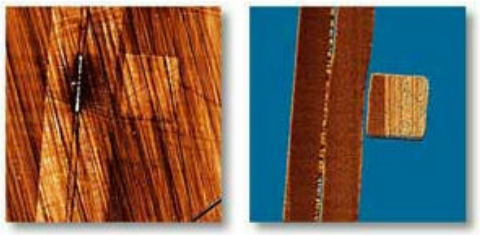
Which effect dominates depends on the tip to surface distance, because magnetic forces persist for greater tip-to-sample separations than van der Waals forces.

If the tip is close to the surface in the non-contact AFM region the image will be predominantly topographic.

As the separation between the tip and the sample increases, magnetic effects become more important. Collecting a series of images at different tip heights we can separate magnetic from topographic effects.



MFM Examples

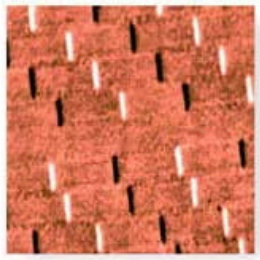


Topography (left) and Magnetic Force Microscopy (right) images

Field of view 15 µm x 15 µm

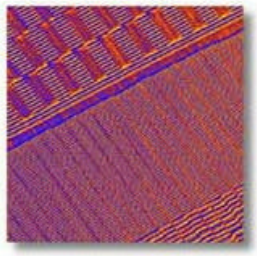
Atomic Force Microscopy (AFM) has been shown to be a valuable tool for measuring the surface morphology of magnetic heads. AFM reveals the morphology of an MR head such as scratches, wear, and contamination and measures pole-tip recession to sub-0.1nm resolution. This resolution will be needed as fly heights decrease. The topographic image reveals the polishing mark and, only slightly, the raised region of the head element. The scratches are as deep as 70 Angstroms, whereas the raised pole region is only 22 Angstroms higher than the background.

Using phase imaging techniques, an MFM image was obtained simultaneously with the topography. This image clearly reveals the domains in the MR head. This MFM image can be used to measure the quality and uniformity of the head.



Field of view 30 µm x 30 µm

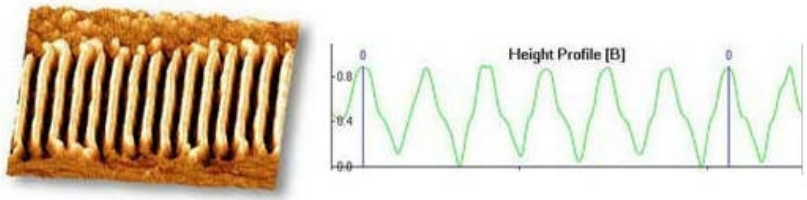
This MFM image clearly shows the bit transition on a disk. Measurements are made of the transition width and straightness. Such measurements can be used to characterize "bit jitter" and are becoming more common. Rice et. al. (to be published) have shown that these images can be compared directly with read-back signals of the same area on the disk to determine the measured micromagnetic data's effect on signal-to-noise of the read-back. This technique is well suited for measuring nonlinear transition shifts and media noise. Therefore, MFM images can be directly related to the output of an inductive record head

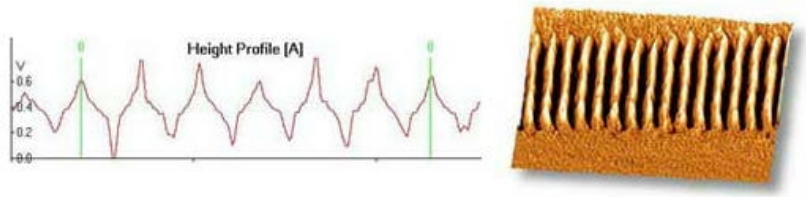


The bright and dark lines indicate transition between the longitudinal bits

Field of view 100 µm x 100 µm

Magnetic force microscopy image of magnetic domains in the servo tracks of a hard disk.





Unkeepered sample (top) and Keepered sample (bottom) MFM images

Field of view 8 µm x 8 µm

These MFM images show a 92 kFCI recording on keepered and unkeepered sides of a disk using a flying inductive thin film head where the keepered side of the disk has a 70nm thick keepered layer. A Co coated MFM tip was used to measure the leakage field from the partially saturated keeper layer. These images show a reduction of the side written signals due to flux fringing during the writing of the keepered surface. In addition, there is evidence that the flux from the transitions on the keepered surface is narrower than that of the unkeepered surface. Cross sections through the bit pattern reveal a narrowing of the transition flux for the keepered layer. This same measurement technique was used for keeper layers as thick as 140nm. This is another example of MFM measurement of micromagnetic domains below the surface.

Scanning Capacitance Microscopy (SCM)

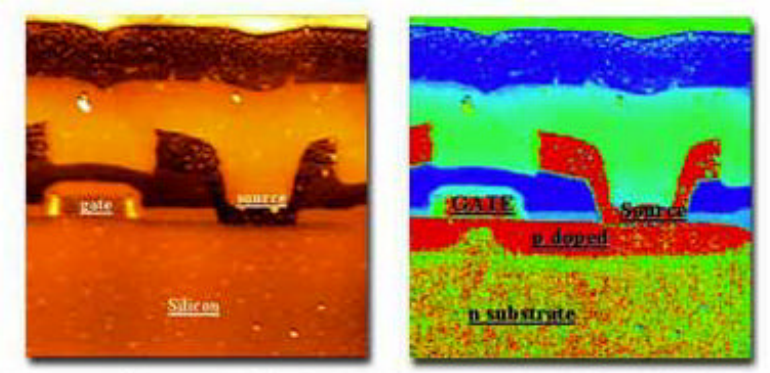
Applications

Dopant profile in semiconductors

Control of mask alignment

Transistor gates

Oxide thickness measurements



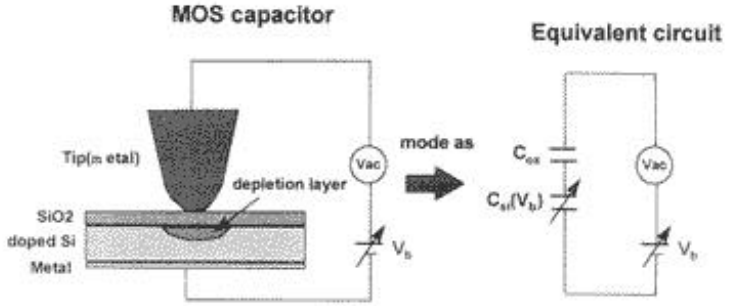
This example shows a depletion transistor with source and gate. The depletion layers are shown and can be measured under the gate using electrostatic force microscopy.

Scanning capacitance microscopy (SCM) images spatial variations in "capacitance".

In SCM voltage is induced between the tip and the sample. The cantilever operates in contact and the capacitance between tip and sample is measured.

Since capacitance depends on the dielectric constant of the medium between the tip and sample, SCM can image variations in the thickness of a dielectric material on a semiconductor substrate.

For example, SCM can be used to visualize sub-surface charge-carrier distributions.



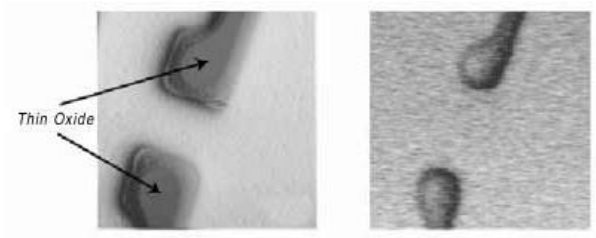
A metallic tip is used to image a semiconductor with its native oxide sample in contact mode AFM. The tip/sample junction becomes a MOS junction.

When a DC bias is applied on the sample, a depletion layer below the semiconductor-insulator interface is created.

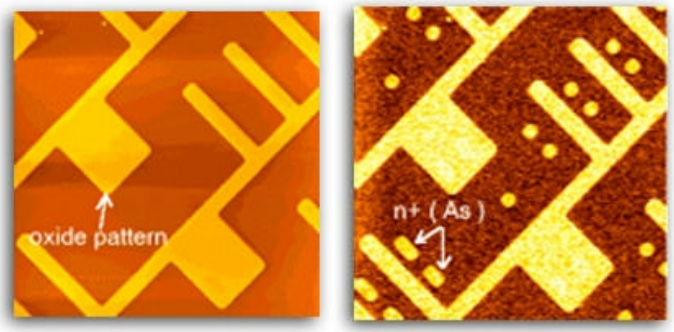
This depletion layer "increases" the oxide thickness of the MOS structure and therefore modifies the capacitance of the junction. Because the measured signal is very small, a lock-in technique is used to improve the signal to noise ratio. The actual measured signal is the dC/dV.

SCM Examples

Transistor Oxide Thickness



Topography (left) of a partially proceed 0.5µm transistor imaged by AFM. SCM image (right) of the same area. The SCM signal is large on the thin oxide and clearly reveals the non-uniformity of the oxide thickness

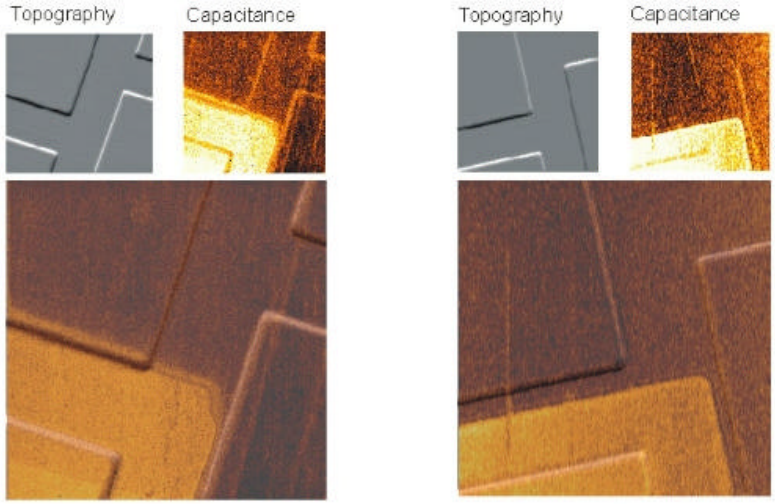


Field of view 70µm x 70µm

One of most common applications of SCM is mapping of the carrier concentration on nonuniformly doped semiconductor samples. Up until now, conventional tools such as SIMS , SRP, and one-dimensional C-V provide dopant or carrier concentration information with very high accuracy and resolution in only one-dimension. Thus quantitative two-dimensional information could be inferred from one-dimensional measurement. However, SCM has shown great potential for direct measurement of two-dimensional activated carrier concentration with nanometer scale accuracy in two-dimensions. The images above represent topography (left) and SCM (right)images of a semiconductor surface.

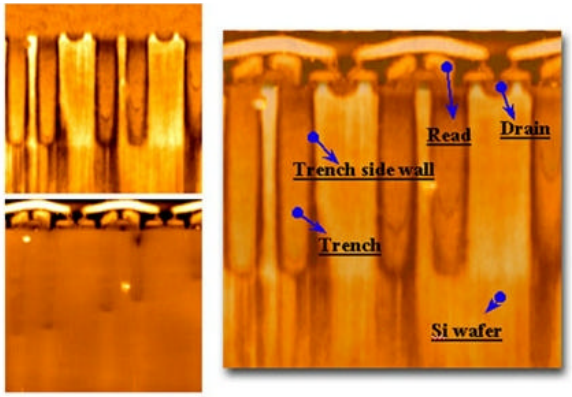
The bright region in the topography image (left) represents thermally grown silicon dioxide pattern with 70 nm height. SCM visualizes and determines the impurity concentration and variation in oxide thickness on the surface. Bright circular and rounded rectangular regions in the SCM image are heavily doped by As+ ion with having 50 keV energy and 10^{14} ions/cm2 dose density. Bright contrast at the patterned oxide region in SCM image indicates that capacitance variation for an ac voltage swing (dC/dV) is very small such as in the heavily doped region due to thick oxide. Therefore this result indicates SCM can measure relative variation in insulator thickness.

Well aligned **Misaligned**



Field of view 30µm x 30µm

The alignment of photo masks during the dopant implantation process is critical for the performance of final semiconductor devices. This example shows two sets of AFM measurements (topography and SCM) for a correctly aligned mask(left) and for a misaligned mask (right). The large pictures are a combination of both the topography (grey) and the SCM image (orange). From these images the amount and direction of the misalignment can be observed.



Field of View 10µm x 10µm

Contact and scanning capacitance images of a DRAM cross-section. Details are revealed by SCM imaging of the trench implants and the drain or source regions of the transistors used to charge the trenches.

Electrostatic Force Microscopy (EFM)

Applications

Failure analysis in microelectronic circuits

Variation of work function of a semiconductor

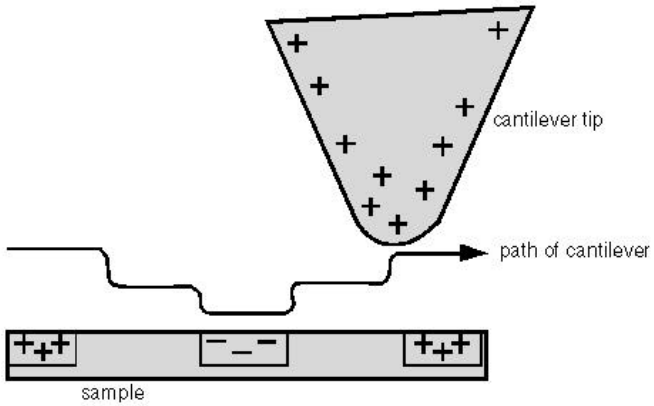
Voltage drop on microresistors

Ferroelectric materials, conductive polymers

Spatial variation of implanted regions

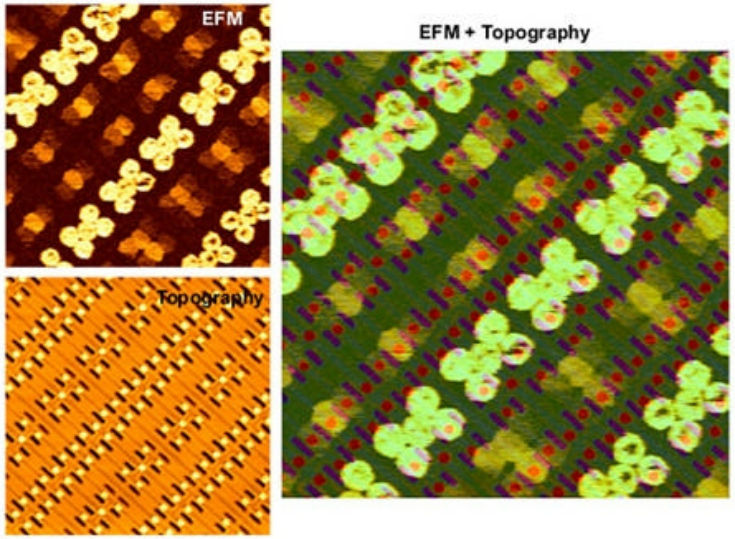
Charge distribution in particles on surfaces

Flat-panel and LCD displays



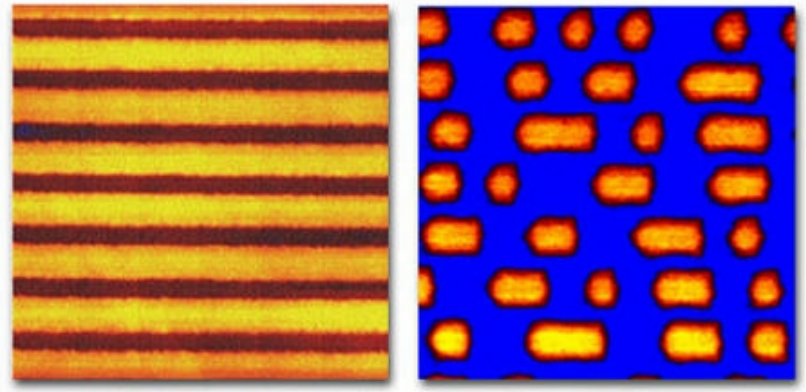
- **EFM allows to image local variations of charge on a surface, or sub-surface to obtain information on buried charge in samples with overcoats, and charge distribution in deposited particles.**
- **EFM can measure variations of near surface dopant levels as a change in sample capacitance and plot this measurement simultaneously with surface topography.**
- **Electrostatic force microscopy applies a voltage between the tip and the sample while the cantilever operates in non-contact mode. The cantilever deflects when it scans over static charges.**
- **EFM maps locally charged domains on the sample surface. The magnitude of the deflection, proportional to the charge density, measured with the standard beam-bounce system.**
- **EFM is used to study the spatial variation of surface charge carrier density.**

EFM Examples



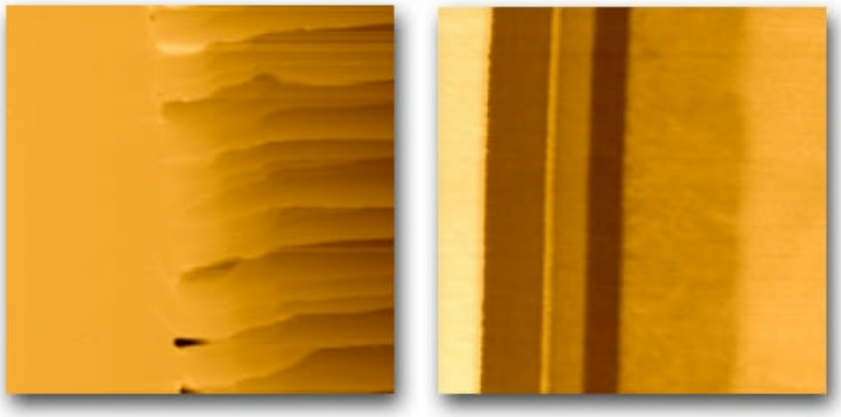
Field of view 20µm x 20µm

EFM (top) can reveal the subsurface dopant profile while topography (bottom) shows the top layers. Merging the EFM and topographical (right) images exposes the mask overlay registration.



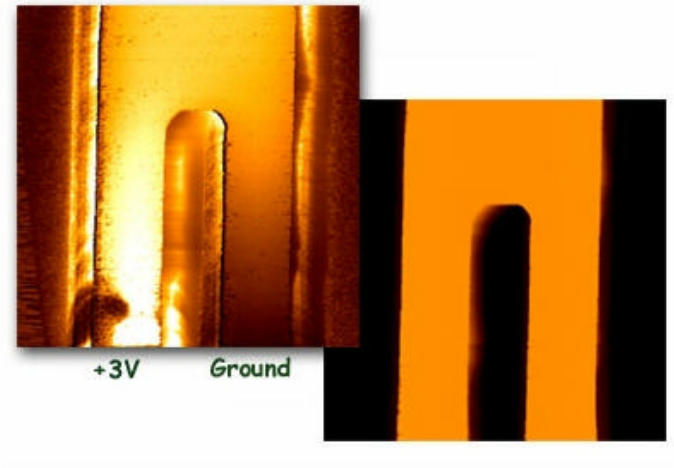
Field of view 5µm x 5µm

Topography (left) and EFM (right) images of a DVD-RW. The EFM image clearly shows amorphous bits formed with the phase change on the crystalline area.



Field of view 10µm x 10µm

Different types of material are deposited on Si wafers during processing. On a polished cross-section, the various materials can be revealed using EFM (right) and the layer thickness can be measured.

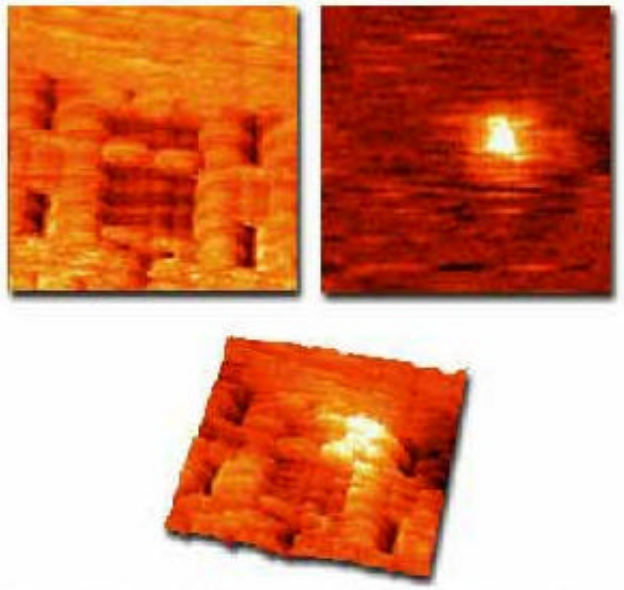


Field of View 70µm x 70µm

EFM, while using 1w, can be used to measure electrostatic potentials on the sample surface. A resistor has been powered with 3 volts, showing the potential drop along the piezolever revealed by using EFM.

Scanning Thermal Microscopy (SThM)

- **SThM can be used in two different operating modes, allowing imaging temperature and thermal conductivity of the sample.**
- **SThM allows simultaneous acquisition of both topographic and thermal conductivity data.**
- **A thermal probe with a resistive element is used.**
- **A cantilever is composed of two different metals (or a thermal element made up of two metal wires.)**



Topographic (upper left) and Thermal (upper right) images of a "hot spot" in a powered IC. The images were added together to get a composite image (bottom)which indicates the location of the failed region.

- **The materials of the cantilever respond differently to changes in thermal conductivity, and cause the cantilever to deflect. The system generates a SThM image, which is a map of the thermal conductivity, from the changes in the deflection of the cantilever.**
- **A topographic non-contact image can be generated from changes in the cantilever's amplitude of vibration. Thus, topographic information can be separated from local variations in the sample's thermal properties, and the two types of images can be collected simultaneously.**

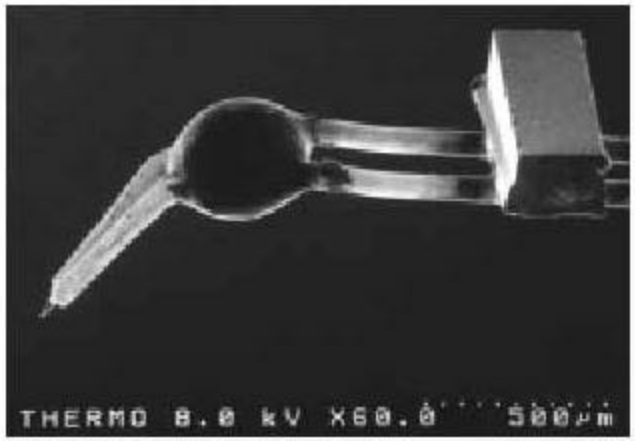
Applications

- **Sub-surface defects, semiconductor failure analysis, conductivity differences in copolymers, surface coatings, etc.**

Scanning Thermal Microscopy (SThM)

A type of thermal cantilever uses a Wollaston wire as the probe. This incorporates a resistive thermal element at the end of the cantilever. The arms of the cantilever are made of silver wire.

The resistive element at the end, which forms the thermal probe is made from platinum or platinum/rhodium 90/10 alloy. The advantage of this design is that it may be used in one of two modes allowing thermal imaging of sample temperature and thermal conductivity.



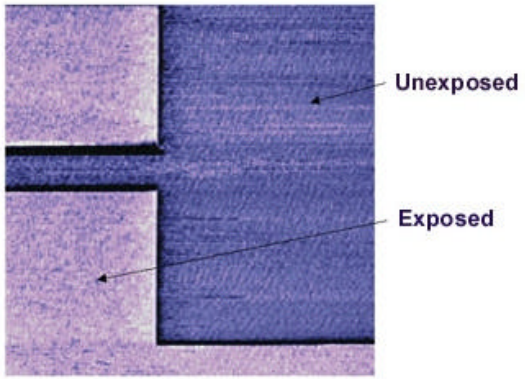
Temperature Contrast Mode

In the temperature contrast mode, the thermal probe is used as a resistance thermometer. Temperature is monitored using a bridge circuit to measure the probe resistance. The probe current is constant and the changes in the probe resistance during scanning reflect the surface temperature changes on the sample surface.

Conductivity Contrast Mode

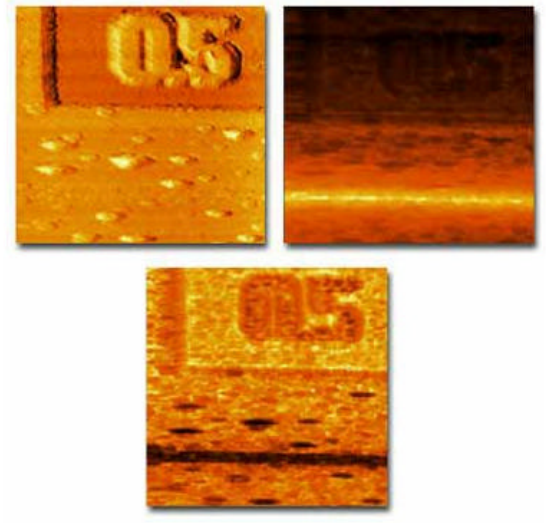
In the conductivity contrast mode, the thermal probe is kept at a constant temperature. The probe temperature remains constant by a feedback loop that keeps the probe resistance constant. Changes in sample thermal conductivity affect the heat flow between the self-heating probe and the sample. This heat flow is monitored, as thermal conductivity, by measuring the voltage necessary to keep the probe at a constant temperature.

SThM Examples



Field of view 100µm x 100µm

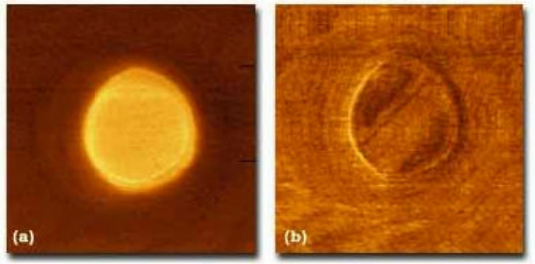
Thermal conductivity of TOK 010 photoresist displaying the exposed and unexposed regions of a device.



Field of view 55µm x 55µm

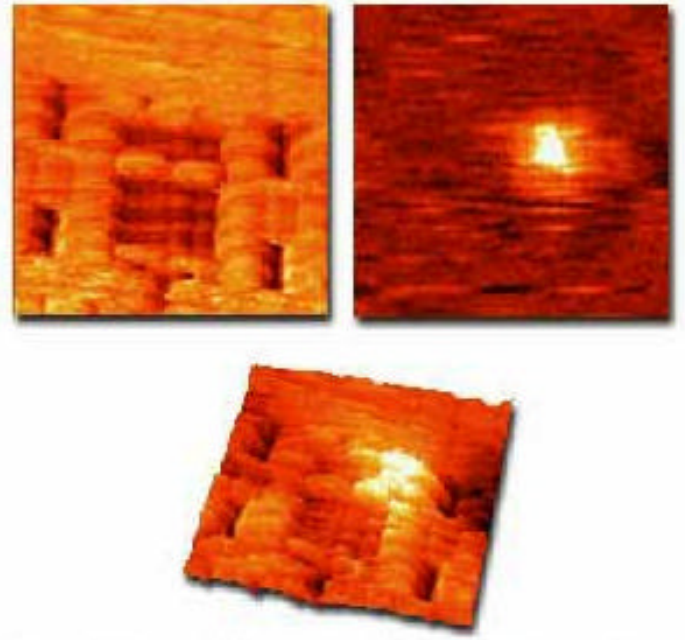
All three of these images were taken simultaneouly using topography (top left) , constant current (top right) , and constant temperature (bottom) modes showing a heated metal trace in semiconductor device. 5V were applied to the heated metal trace.

Depth Profiling



Field of view 100µm x 100µm

Two ac-thermal images of a sample with an island of high-thermal-conductivity material within a matrix of low-thermal-conductivity material, over both of which there is a polymer coating. Image (a) was taken at 1 kHz and (b) at 30kHz.



Topographic (upper left) and Thermal (upper right) images of a "hot spot" in a powered IC. The images were added together to get a composite image (bottom)which indicates the location of the failed region.