

### A Practical Guide to

## SPM

**Scanning Probe Microscopy** 



#### A Practical Guide to SPM

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#### I. Introduction

In the early 1980s, scanning probe microscopes (SPMs) dazzled the world with the first real-space atomic-scale images of surfaces. Now, SPMs are used in a wide variety of disciplines, including fundamental surface science, routine surface roughness analysis, and spectacular three-dimensional imaging — from atoms of silicon to micron-sized protrusions on the surface of a living cell.

The scanning probe microscope is an imaging tool with a vast dynamic range, spanning the realms of optical and electron microscopes. It is also a profiler with unprecedented resolution. In some cases, scanning probe microscopes can measure physical properties such as surface conductivity, static charge distribution, localized friction, magnetic fields, and elastic moduli. Hence, SPM applications are very diverse.

This guide was written to help you learn about SPMs, a process that should begin with a thorough understanding of the basics. Issues covered in this guide range from fundamental physics of SPMs to practical capabilities and instrumentation. Examples of applications are included throughout.

The origins of Veeco SPMs go back to the late 1980s. Since that time, we have maintained strong ties to the academic community and a corporate philosophy that combines technology leadership with a practical-applications orientation, working with customers to demonstrate the ability of our SPMs to meet their needs. We believe that the more you know about scanning probe microscopes, the more likely you will be to choose the best instrument for your work. We want to provide you with the basic facts about SPMs before you make your way through sales literature.

#### II. How an SPM Works

Scanning probe microscopes are a family of instruments used for studying surface properties of materials from the micron all the way down to the atomic level. Two fundamental components that make scanning probe microscopy possible are the **probe** and the **scanner**. The probe is the point of interface between the SPM and the sample; it is the probe that intimately interrogates various qualities of the surface. The scanner controls the precise position of the probe in relation to the surface, both vertically and laterally.

#### The Probe

When two materials are brought very close together, various interactions are present at the atomic level. These interactions are the basis for scanning probe microscopy. An SPM probe is a component that is particularly sensitive to such interactions and is designed to sense them. Specifically, when an SPM probe is brought very close to

Means of sensing the vertical position of the tip

A feedback system to control the vertical position of the tip

A probe tip

A probe tip

A probe tip into the general vicinity of the sample

A piezoelectric scanner that moves the sample under the tip (or the tip over the sample) in a raster pattern

A computer system that drives the scanner, measures data, and converts the data into an image

Figure 2-1. SPM feedback loop.

a sample surface, the sensed interaction can be correlated to the distance between the probe and sample. Since the magnitude of this interaction varies as a function of the probe-sample distance, the SPM can map a sample's surface topography by scanning the probe in a precise, controlled manner over the sample surface.

#### The Scanner

The material that provides the precise positioning control required by all SPM scanners is piezoelectric ceramic. Piezoelectric ceramic changes its geometry when a voltage is applied; the voltage applied is proportional to the resulting mechanical movement.

The piezoelectric scanner in an SPM is designed to bend, expand, and contract in a controlled, predictable manner. The scanner, therefore, provides a way of controlling the probe-sample distance and of moving the probe over the surface.

#### Scanning: Setpoint, Detector Signal, and Error Signal

In order to generate an SPM image, the scanner moves the probe tip close enough to the sample surface for the probe to sense the probesample interaction. Once within this regime, the probe produces a signal representing the magnitude of this interaction, which corresponds to the probe-sample distance. This signal is referred to as the **detector signal (Figure 2-1).** 

In order for the detector signal to be meaningful, a reference value known as the **setpoint** is established. When the scanner moves the probe into the imaging regime, the detector signal is monitored and compared to the setpoint. When the detector signal is equal to the setpoint, the scanning can begin.

The scanner moves the probe over the surface in a precise, defined pattern known as a raster pattern, a series of rows in a zigzag pattern covering a square or rectangular area. As the probe encounters changes in the sample topography, the probe-sample distance changes, triggering a corresponding variance in the detector signal. The data for generating an SPM image is calculated by comparing the detector signal to the setpoint. The difference between these two values is referred to as the **error signal**, which is the raw data used to generate an image of the surface topography.

Data can be collected as the probe moves from left to right (the "trace") and from right to left (the "retrace"). The ability to collect data in both directions can be very useful in factoring out certain effects that do not accurately represent the sample surface. The trace-and-retrace movement is sometimes referred to as the "fast scan direction." The direction perpendicular to the fast scan direction is sometimes referred to as the "slow scan direction."

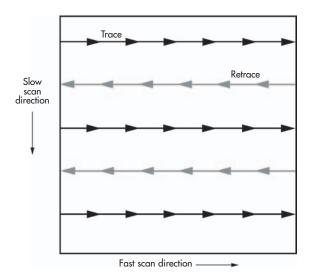


Figure 2-2. Trace and retrace.

**Figure 2-2** shows scan lines parallel. In reality, they form a zigzag pattern in which the end of each scan line to the left is identical to the beginning of the next scan line to the right, and vice versa. Typically, the scan lines covering the square or rectangular scan area are numerous enough that consecutive scan lines in opposite directions (trace and retrace) are approximately parallel.

#### The SPM Image

As the probe scans each line in the raster pattern, the error signal can be interpreted as a series of data points. The SPM image is then generated by plotting the data point-by-point and line-by-line. Other signals can also be used to generate an image.

SPM imaging software displays the image in a useful way. For example, the height and color scales can be adjusted to highlight features of interest. The number of data points in each scan line and the number of scan lines that cover the image area will determine the image resolution in the fast and slow scan directions, respectively.

#### The Z Feedback Loop

SPMs employ a method known as **Z feedback** to ensure that the probe accurately tracks the surface topography. The method involves continually comparing the detector signal to the setpoint. If they are not equal, a voltage is applied to the scanner in order to move the probe either closer to or farther from the sample surface to bring the error signal back to zero. This applied voltage is commonly used as the signal for generating an SPM image.

Scanning can be performed with Z feedback turned on or off. With feedback off, the error signal is used to generate the image. With feedback on, the image is based on the voltage applied to the scanner. Each mode has advantages and disadvantages. Scanning with Z feedback turned off is faster, as the system does not have to move the scanner up and down, but it only provides useful measurement information for relatively smooth surfaces. While scanning with feedback on takes more time, it allows the measurement of irregular surfaces with high precision.

#### Scanning Tunneling Microscopy (STM)

Scanning tunneling microscopy (STM) measures the topography of a surface using a tunneling current that is dependent on the separation between the probe tip and the sample surface. STM is typically performed on conductive and semiconductive surfaces. Common applications include atomic resolution imaging, electrochemical STM, scanning tunneling spectroscopy (STS), and low-current imaging of poorly conductive samples using low-current STM.

The scanning tunneling microscope (STM) is the ancestor of all scanning probe microscopes. Invented in 1981 at IBM Zurich, it was the first instrument to generate real-space images of surfaces with atomic resolution. The inventors, Gerd Binnig and Heinrich Rohrer, were awarded the Nobel Prize in Physics five years later for this invention.

The probe in an STM is a conducting sharp tip (typically made of platinum-iridium or tungsten). A bias voltage is applied between the tip and the sample. When the tip is brought close enough to the sample, electrons begin to "tunnel" through the gap, from the tip into the sample, or vice versa, depending on the polarity of the bias voltage. The resulting tunneling current varies with tip-sample distance and is the detector signal used to create an STM image. Note that for tunneling to take place, both the sample and the tip must be conductors or semiconductors. Unlike AFMs, which are discussed in the next section, STMs cannot image insulating materials.

The tunneling current is an exponential function of the distance such that in a typical experiment if the separation between the tip and the sample changes by about an Ångstrom, the tunneling current changes by an order of magnitude. This gives the STM its remarkable sensitivity. The STM can image the surface of a sample with atomic resolution — vertically and laterally.

#### **Atomic Force Microscopy (AFM)**

The atomic force microscope (AFM) grew out of the STM and today it is by far the more prevalent of the two. Unlike STMs, AFMs can be used to study insulators, as well as semiconductors and conductors.

The probe used in an AFM is a sharp tip, typically less than 5µm tall and often less than 10nm in diameter at the apex. The tip is located at the free end of a cantilever that is usually 100–500µm long. Forces between the tip and the sample surface cause the cantilever to bend, or deflect. A detector measures the cantilever deflections as the tip is scanned over the sample, or the sample is scanned under the tip. The measured cantilever deflections allow a computer to generate a map of surface topography. Several forces typically contribute to the deflection of an AFM cantilever. To a large extent, the distance regime (i.e., the tip-sample spacing) determines the type of force that will be sensed.

Variations on this basic scheme are used to measure topography as well as other surface features. There are numerous AFM modes. Each is defined primarily in terms of the type of force being measured and how it is measured.

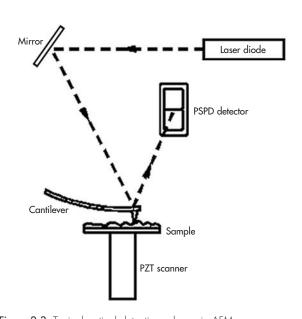


Figure 2-3. Typical optical detection scheme in AFM.

Most AFMs use optical techniques to detect the position of the cantilever. In the most common scheme (Figure 2-3), a light beam from a laser diode bounces off the back of the cantilever and onto a position-sensitive photo-detector (PSPD). As the cantilever bends, the position of the laser beam on the detector changes. The ratio of the path length between the cantilever and the detector to the length of the cantilever itself produces amplification. As a result, the system can detect sub-Ångstrom vertical movement at the free end of the cantilever, where the tip is located.

Once the AFM has detected the cantilever deflection, it can generate the topographic data with the Z feedback turned on or off. With Z feedback off (constant-height mode), the spatial variation of the cantilever deflection is used to generate the topographic data set. With Z feedback on (constant-force mode), the image is based on the Z motion of the scanner as it moves in the Z direction to maintain a constant cantilever deflection.

In constant-force mode, the speed of scanning is limited by the response time of the feedback loop, 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 essential for recording real-time images of changing surfaces, where high scan speed is a must.

## III. Near-field Scanning Optical Microscopy (NSOM)

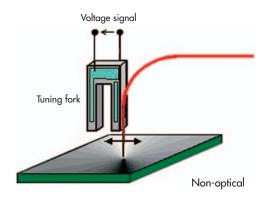


Figure 3-1. Tuning fork mechanism.

NSOM is an optical microscopy technique that takes advantage of SPM technology to enable users to work with standard optical tools beyond the diffraction limit that normally restricts the resolution capability of such methods. NSOM works by exciting the sample with light passing through a sub-micron aperture formed at the end of a single-mode drawn optical fiber. Typically, the aperture is a few tens of nanometers in diameter. The fiber is coated with aluminum to prevent light loss, thus ensuring a focused beam from the tip.

As in SPMs, a probe measures the tip-sample distance and piezoelectric scanners are used to scan the sample in a defined pattern and respond to changes in the sample topography. These two technologies make it possible to bring the aperture of the optical fiber into the "near-field" regime and maintain that distance throughout the scanning process. In NSOM, the probe may be tuning fork-based shear-force feedback. Tuning fork technology eliminates the need for an additional feedback laser, as found in earlier NSOM designs (Figure 3-1).

Tuning forks also improve force sensitivity and greatly facilitate setup, thus allowing a broader choice of light wavelengths for measurement. Simultaneous, independent optical and topographic images can then be taken. It is important that only a very small portion of the sample be illuminated. This significantly reduces photobleaching, which helps clarify the relationship between optical and topographic structure.

Fluorescence is the most commonly used imaging mode; other modes include UV-visible, IR, and Raman techniques. In addition to its remarkable imaging capabilities, chemical information can be obtained using near-field spectroscopy at resolutions better than 100nm.

### IV. Primary AFM Imaging Modes

This chapter discusses the four primary AFM modes: TappingMode, contact, non-contact, and torsional resonance. Many secondary modes (which will be described in the next chapter) can be derived from these primary modes.

#### TappingMode AFM

TappingMode<sup>TM</sup> AFM, the most commonly used of all AFM modes, is a patented technique (Veeco Instruments) that maps topography by lightly tapping the surface with an oscillating probe tip. The cantilever's oscillation amplitude changes with sample surface topography. The topography image is obtained by monitoring these changes and closing the Z feedback loop to minimize them.

TappingMode has become an important AFM technique, as it overcomes some of the limitations of both contact and non-contact AFM (also described in this chapter). By eliminating lateral forces that can damage samples and reduce image resolution, TappingMode allows routine imaging of samples once considered impossible to image with AFMs.

Another major advantage of Tapping/Mode is related to limitations that can arise due to the thin layer of liquid that forms on most sample surfaces in an ambient imaging environment (i.e., in air or some other gas). The amplitude of the cantilever oscillation in Tapping/Mode is typically on the order of a few tens of nanometers, which ensures that the tip does not get stuck in this liquid layer. The amplitude used in non-contact AFM is much smaller. As a result, the non-contact tip often gets stuck in the liquid layer unless the scan is performed at a very slow speed.

In general, TappingMode is much more effective than non-contact AFM, but especially for imaging larger areas on the sample that may include greater variation in topography. TappingMode can be performed in gases and non-corrosive liquids.

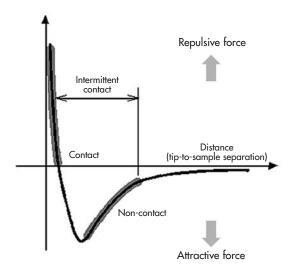


Figure 4-1. Attractive and repulsive forces.

#### **Contact AFM**

In contact AFM, the tip is in perpetual 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 of the force holding the atoms of most solids together. As the scanner gently traces the tip across the sample (or the sample under the tip), the contact force causes the cantilever to bend to accommodate changes in topography.

At the right side of the curve in Figure 4-1, the tip and sample are separated. As the tip and the sample are gradually brought together, their atoms begin to weakly attract each other. This attraction increases until the atoms are so close together that their electron clouds begin to repel each other. This electrostatic repulsion progressively weakens the attractive force as the separation continues to decrease. The total force goes through zero and finally becomes positive (repulsive).

The slope of the curve is very steep in the repulsive regime. This force balances and surpasses 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 use a very stiff cantilever to exert large forces on the sample, the inter-atomic separation between the tip and sample atoms does not decrease much. Instead, the sample surface, or the tip, or both, are likely to deform.

In addition to the repulsive force described above, two other forces are generally present during contact AFM imaging: a capillary force exerted by the thin liquid layer often present on the sample surface in an ambient (non-vacuum) environment (typically, this layer is mostly water), and the force exerted by the cantilever itself. The capillary force arises when water wicks its way around the tip, applying a strong attractive force that tends to hold the tip in contact with the surface. The magnitude of the capillary force, too, depends upon the tip-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. These forces are revisited in the Non-imaging Modes chapter.

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. (We assume here that the water layer is homogeneous across the scanning area.) The variable force in contact AFM is then the force exerted by the cantilever.

# Angstrom-scale lateral tip dither

Figure 4-2. TRmode.

#### **Non-contact AFM**

Non-contact AFM (NC-AFM) is one of several vibrating cantilever techniques in which an AFM cantilever is vibrated near the surface of a sample. The spacing between the tip and the sample for NC-AFM, on the order of tens to hundreds of Ångstroms, is indicated in **Figure 4-1** as the non-contact regime. The inter-atomic force between the cantilever and sample in this regime is attractive (largely a result of van der Waals interactions).

In non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically 100–400 kHz) with amplitude of a few tens to hundreds of Ångstroms. The system detects changes in the cantilever's resonance frequency or vibration amplitude. The attractive non-contact force between tip and sample is typically smaller than and more difficult to maintain than the repulsive contact force. In addition, cantilevers used for NC-AFM must be stiffer than those used for contact AFM. These are both factors that make the NC-AFM detector signal small. Therefore, a sensitive AC detection scheme is used for NC-AFM imaging.

In NC-AFM mode, Z feedback is turned on. The system monitors either the resonant frequency or vibration amplitude of the cantilever and keeps it constant by moving the scanner up or down in response to changes. In this way, the average tip-sample distance is also kept constant and the Z feedback signal is used to generate the data set.

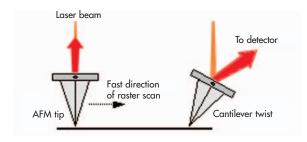
#### **Torsional Resonance Mode (TRmode) AFM**

TRmode $^{\mathsf{TM}}$  is a major new patented technique (Veeco Instruments) in atomic force microscopy that measures and controls dynamic lateral forces between the AFM probe tip and sample surface. Utilizing advanced sensing hardware and electronics to characterize torsional oscillations of the cantilever, TRmode enables detailed, nanoscale examination of in-plane anisotropy and provides new perspectives in the study of material structures and properties. It can also be interleaved with TappingMode to provide complementary lateral and vertical characterization.

In TRmode, the cantilever oscillates around its long axis in a twisting motion. This oscillation causes a dithering motion of the tip. As the tip encounters lateral forces on the sample surface, the corresponding change in the cantilever's twisting motion is detected (the detector signal).

This twist is detected by using a quad-cell position-sensitive photodetector (PSPD). With a quad-cell PSPD, vertical deflection and lateral twist can be measured simultaneously, as shown in **Figure 4-2**. Note that contact AFM uses a bi-cell PSPD to measure the vertical deflection of the cantilever, indicating changes in sample topography.

#### V. Secondary AFM Imaging Modes



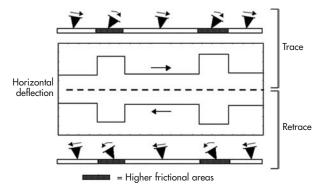


Figure 5-1. Lateral force microscopy.

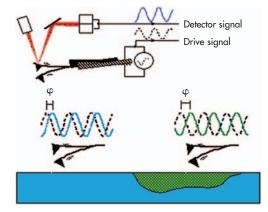


Figure 5-2. Phase imaging.

The imaging modes described below are derived from the four primary AFM imaging modes discussed in the previous chapter.

#### **Lateral Force Microscopy**

Lateral force microscopy (LFM) is a contact AFM mode that identifies and maps relative differences in the friction forces between the probe tip and the sample surface. In LFM, the scanning is always perpendicular to the long axis of the cantilever. Forces on the cantilever that are parallel to the plane of the sample surface cause twisting of the cantilever around its long axis. This twisting is measured by a quad-cell PSPD, as with TRmode.

Twisting of the cantilever usually arises from two sources: changes in surface friction and changes in topography, as shown in Figure 5-1. In the first case, the tip may experience greater friction as it traverses some areas, causing the cantilever to twist more. In the second case, the cantilever may twist when it encounters edges of topographic features. To separate one effect from the other, usually three signals are collected simultaneously: both trace and retrace LFM signals, along with either the trace or retrace AFM signal.

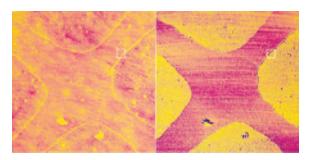
LFM applications include identifying transitions between different components in polymer blends and composites, identifying contaminants on surfaces, delineating coverage by coatings, and chemical force microscopy (CFM) using probe tips functionalized for specific chemical species.

#### **Phase Imaging**

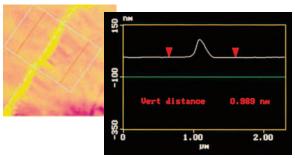
Phase imaging is the mapping of the measure phase of the cantilever's periodic oscillations, relative to the phase of the periodic signal that drives the cantilever. Changes in this measured phase often correspond to changes in the properties across the sample surface (Figure 5-2).

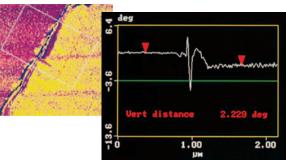
Phase imaging is a secondary imaging mode derived from TappingMode or non-contact mode that goes beyond topographic data to detect variations in composition, adhesion, friction, viscoelasticity, and other properties, including electric and magnetic. Applications include locating contaminants, mapping different components in composite materials, and differentiating regions of high and low surface adhesion or hardness as well as regions of different electrical or magnetic properties.

The AFM's feedback loop operates in the usual manner, using changes in the cantilever's oscillation amplitude to map sample topography. The phase is monitored while the topographic image is being taken so that images of topography and phase (material properties) can be collected simultaneously.

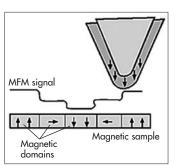


**Figure 5-3.** Simultaneously acquired topography (left) and phase (right) AFM images of a silicone hydrogel in saline solution. Field of view: 50µm.





**Figure 5-4.** Detail of Figure 5-3, showing boundary between hydrophilic and hydrophobic regions in topography (top) and phase (bottom) images.



**Figure 5-5.** MFM maps the magnetic domains of the sample surface.

Figure 5-6. MFM image showing the magnetic signature of data bits written on a hard disk, 3µm scan.



**Figure 5-3** shows simultaneously acquired topography and phase AFM images of a silicone hydrogel in saline solution. The four outer areas were exposed to a sequence of chemical processing steps. The central cross-like region, which was masked and thus protected from the processing, retained its hydrophobicity.

In the phase image (Figure 5-4), a marked phase shift is seen across the boundaries. The phase image is clearly providing material property contrast on this well-defined experimental hydrogel surface.

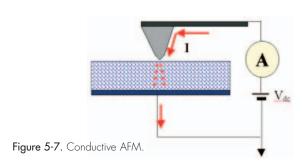
The phase signal is sensitive to both short- and long-range tip-sample interactions. Short-range interactions include adhesive forces and frictional forces; long-range interactions include electric fields and magnetic fields.

Phase detection is a key element in numerous scanning probe microscopy techniques, including magnetic force microscopy (MFM), electric force microscopy (EFM), and scanning capacitance microscopy (SCM).

#### **Magnetic Force Microscopy**

Magnetic force microscopy (MFM) is a secondary imaging mode derived from TappingMode that maps magnetic force gradient above the sample surface. This mapping is performed via a patented two-pass technique, LiftMode. LiftMode separately measures topography and another selected property (magnetic force, electric force, etc.) using the topographical information to track the probe tip at a constant height above the sample surface during the second pass.

The MFM probe tip is coated with a ferromagnetic thin film. While scanning, it is the magnetic field's dependence on tip-sample separation that induces changes in the cantilever's amplitude, resonance frequency, or phase (Figure 5-5). MFM can be used to image both naturally occurring and deliberately written magnetic domains (Figure 5-6).



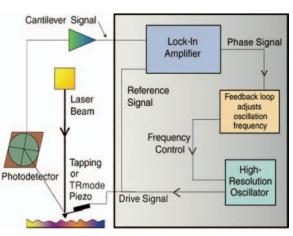


Figure 5-8. Electric force microscopy.

#### **Conductive AFM**

Conductive atomic force microscopy (CAFM) is a secondary imaging mode derived from contact AFM that characterizes conductivity variations across medium to low-conducting and semiconducting materials. CAFM performs general-purpose measurements and has a current range of pA to  $\mu$ A.

CAFM employs a conductive probe tip. Typically, a DC bias is applied to the tip and the sample is held at ground potential. While the Z feedback signal is used to generate a normal contact AFM topography image, the current passing between the tip and sample is measured to generate the conductive AFM image (Figure 5-7). Alternatively, using a second feedback loop, the applied voltage is modified to yield a constant current. It is the voltage data that constitutes the image.

#### **Tunneling AFM (TUNA)**

Tunneling AFM (TUNA) works similarly to conductive AFM, but with higher sensitivities. TUNA characterizes ultra-low currents (between 80fA and 120pA) through the thickness of thin films. The TUNA application can be operated in either imaging or spectroscopy mode (see the Non-imaging Modes chapter). Applications include gate dielectric development in the semiconductor industry.

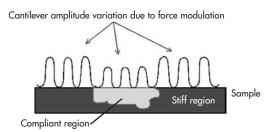
#### **Electric Force Microscopy**

Electric force microscopy (EFM) is a secondary imaging mode derived from TappingMode that measures electric field gradient distribution above the sample surface. The measurement is performed via LiftMode, a patented two-pass technique mentioned earlier in this chapter. LiftMode separately measures topography and another selected property (magnetic force, electric force, etc.) using the topographical information to track the probe tip at a constant height above the sample surface during the second pass.

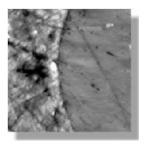
Often, a voltage is applied between the tip and the sample in EFM. (Sometimes this voltage is not required to obtain an EFM image.) The cantilever's resonance frequency and phase both change with the strength of the electric field gradient and are used to construct the EFM image. Locally charged domains on the sample surface are mapped in a manner similar to the way in which MFM maps magnetic domains (Figure 5-8).

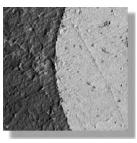
#### **Surface Potential Imaging**

Surface potential (SP) imaging is a secondary imaging mode derived from TappingMode that maps the variation of the electrostatic potential across the sample surface. SP imaging is a nulling technique. As the tip travels above the surface in LiftMode (see the preceding section on EFM), the tip and the cantilever experience a force wherever the



**Figure 5-9.** Force modulation imaging. The amplitude of cantilever oscillation varies according to the mechanical properties of the sample surface.





**Figure 5-10.** Contact AFM (left) and force modulation (right) images of a carbon fiber/polymer composite collected simultaneously, 5µm scans.

potential on the surface is different from the potential of the tip. The force is nullified by varying the voltage of the tip so that the tip is at the same potential as the region of the sample surface underneath it. SP imaging can be used to detect and quantify contact potential differences (CPD) on the surface. SP imaging has found numerous applications, including corrosion science.

#### **Force Modulation Imaging**

Force modulation imaging is a secondary imaging mode derived from contact AFM that measures relative elasticity/stiffness of surface features. It is commonly used to map the distribution of materials of composite systems. As with LFM and MFM, force modulation imaging allows simultaneous acquisition of both topographic and material-properties maps.

In force modulation imaging mode, the probe tip tracks the sample topography as in normal contact AFM. In addition, a periodic mechanical signal, typically less than 5 kHz, is applied to the base of the cantilever. The amplitude of cantilever modulation that results from this applied signal varies according to the elastic properties of the sample, as shown in **Figure 5-9**.

The resulting force modulation image is a map of the sample's elastic response. The frequency of the applied signal is typically tens of kilohertz, which is faster than the Z feedback loop is set up to track. Thus, topographic information can be separated from local variations in the sample's elastic properties and the two types of images can be collected simultaneously, as shown in **Figure 5-10**.

#### **Scanning Capacitance Microscopy**

Scanning capacitance microscopy (SCM) is a secondary imaging mode derived from contact AFM. SCM maps variations in majority electrical carrier concentration (electrons or holes) across the sample surface (typically a doped semiconductor). SCM applies a high-frequency (90 kHz) AC bias to the sample and uses an ultra-high-frequency (1 GHz) detector to measure local tip-sample capacitance changes as the tip scans across the sample surface. These capacitance changes are a function of the majority carrier concentration in semiconductors; hence, relative carrier concentration can be mapped in the range of  $10^{16}$ – $10^{21}$  cm<sup>3</sup>.

Applications include two-dimensional profiling of dopants in semiconductor process evaluation and failure analysis.

# Cantilever mount Wollaston wire Platinum core



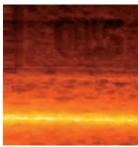
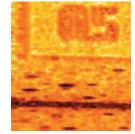


Figure 5-11. Simultaneous acquisition of topography and thermal data. Applied 5V to metal trace, constant current mode (top, right), and constant temperature mode (bottom, right).



#### **Scanning Spreading Resistance Microscopy (SSRM)**

Scanning spreading resistance microscopy (SSRM) is a patented (Veeco Instruments) secondary imaging mode derived from contact AFM that maps variations in majority carrier concentration in semiconductor materials. A conductive probe is scanned in contact mode across the sample, while a DC bias is applied between the tip and sample. The resulting current between the tip and sample is measured (referencing it to an internal resistor) using a logarithmic current amplifier, providing a range of 10pA-0.1mA. This yields a local resistance measurement, which is mapped to generate the SSRM image.

#### **Scanning Thermal Microscopy**

Scanning thermal microscopy (SThM) is a secondary imaging mode derived from contact AFM that maps two-dimensional temperature variation across the sample surface. As with many other modes, SThM allows simultaneous acquisition of topographic data. At the heart of an SThM system is a thermal probe with a resistive element. A thermal control unit carries out thermal mapping and control to produce images based on variations in either sample temperature or thermal conductivity. Common applications include semiconductor failure analysis, material distribution in composites, and magnetoresistive head characterization.

Different types of cantilevers are available for SThM. The cantilever can be composed of two different metals or it can have a thermal element made up of two metal wires. The materials of the cantilever respond differently to changes in thermal conductivity, causing the cantilever to deflect. The system uses the changes in cantilever deflection to generate an SThM image — a map of the thermal conductivity. A topographic 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.

Another type of thermal cantilever uses a Wollaston wire as the probe, which 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/10% rhodium alloy. This design has the advantage of being capable of thermal imaging of either sample temperature or thermal conductivity (Figure 5-11).

#### **VI. Non-imaging Modes**

SPMs are most commonly thought of as tools for generating images of a sample's surface. However, SPMs can also be used to measure material properties at a single X,Y point on the sample surface. (In the SPM literature, this is often referred to as SPM spectroscopy, which we adopt here, and which is quite different from the traditional definition of spectroscopy in the sciences.) SPMs are also used to alter — rather than measure — the sample surface. Examples include nanolithography, nanomanipulation, molecular pulling, and nanoindentation. These non-imaging modes of SPMs are increasingly used in surface science. In this chapter, we describe the most popular nonimaging SPM modes.

#### **Spectroscopy**

#### **Scanning Tunneling Spectroscopy (STS)**

A scanning tunneling microscope (STM) can be used as a spectroscopy tool, probing the electronic properties of a material with atomic resolution in the X-Y plane of the sample surface. This technique is called scanning tunneling spectroscopy (STS). By comparison, traditional spectroscopy techniques like XPS (x-ray photoelectron spectroscopy), UPS (ultraviolet photoelectron spectroscopy), and IPES (inverse photo-emission spectroscopy) detect and average the data originating from a relatively large area: a few microns to a few millimeters across.

STS studies the local electronic structure of a sample's surface. The electronic structure of an atom depends upon its atomic species (e.g., whether it is a gallium atom or an arsenic atom) and its local environment (how many neighboring atoms it has, what kind of atoms they are, and the symmetry of their distribution).

STS encompasses many methods. For example, at constant tip-sample separation, we can ramp the bias voltage with the tip positioned over a feature of interest while recording the tunneling current. This results in current vs. voltage (I-V) curves, which characterize the electronic structure at a specific X,Y location. An STM can be set up to perform STS (collect I-V curves) at numerous points across the sample surface, providing a three-dimensional map similar to force volume imaging in AFM, which is also described in this chapter.

#### **Force Spectroscopy**

An AFM can be used as a spectroscopy tool, probing the tip-sample interaction at a given X,Y location on the sample surface. In SPM literature, this is typically called force spectroscopy because the interaction results in a force (as opposed to a current in STS). This method typically produces a force vs. distance curve, which is a plot of the force on an AFM cantilever (via the tip) in the Z direction as a function of the tip-sample separation in Z. Force vs. distance curves were first used to measure the vertical force the AFM tip applies to the

Force

Distance
Sample

Sample

Lube

C1

Lube

C2

Lube

Figure 6-1. Force vs. distance curves in vacuum (top), air (middle), and air with a contamination layer (bottom).

surface in contact AFM imaging. Soon, this technique proliferated and then matured as a separate investigation method. Force spectroscopy can also be used to analyze the adhesion of surface contaminants, as well as local variations in the elastic properties. Other configurations are also used. For example, at a given X,Y location, and a given tip-sample separation in Z, a bias can be applied and ramped between the sample and the tip so that the cantilever deflection is measured in response to the electrical interaction between the tip and the sample.

A force vs. distance curve is a plot of the deflection of the cantilever versus the extension of the piezoelectric scanner that changes the distance between the sample and the tip. Contaminants and lubricants affect force spectroscopy measurements, as do thin layers of adsorbates on the sample surface.

In the laboratory, force-distance curves are quite complex and specific to the given system under study. The graphics here represent a simplification — the shapes, sizes, and distances are not to scale.

Consider the simplest case of an AFM in vacuum. At the top of **Figure 6-1**, on the left side of the curve, the scanner is fully retracted and the cantilever is un-deflected, as the tip and sample are far apart. As the scanner extends, closing the tip-sample gap, the cantilever remains un-deflected until the tip comes close enough to the sample surface to experience the attractive van der Waals force. The cantilever bends slightly towards the surface and then the tip snaps into the surface (point **a**).

As the scanner continues to extend, the cantilever deflects, being pushed by the surface (region **b**). After full extension of the scanner, at the extreme right of the plot, the scanner begins to retract. The cantilever deflection retraces the same curve, in the reverse direction.

In air, the retracting curve is often different because a monolayer or a few monolayers of water are present on many surfaces (Figure 6-1, middle). This water layer exerts an attractive capillary force on the tip. 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, the scanner retracts enough so that the tip snaps free from the surface (point d). As the scanner continues to retract, the cantilever, now free, remains un-deflected.

If a lubrication or contamination layer is present on the sample surface, multiple tip-snaps can occur (**Figure 6-1**, bottom). The positions and amplitudes of the snaps depend upon the adhesion and thickness of the layers present on the surface.

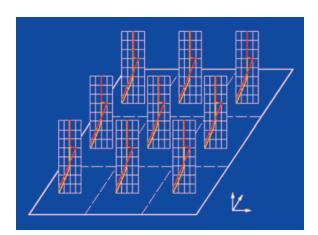


Figure 6-2. Force volume.

In the linear region of the curves (region  $\mathbf{b}$ ), the slope is related to the cantilever stiffness when the sample is infinitely hard. Here, the slope of the curve reflects the spring constant of the cantilever. However, when the cantilever is stiffer than the sample surface, or nearly as stiff as the sample surface, then the slope of the curve may enable investigation of the elastic properties of the sample.

Force spectroscopy can also be done with an oscillating cantilever. In this case, we can plot the cantilever's amplitude, or phase, or both, versus the ramped parameter, which typically is the scanner voltage (tip-sample separation).

#### **Force Volume**

Force volume imaging is a method for studying correlations between tip-sample forces and surface features by collecting a data set containing both topographic data and force-distance curves. As the topographic data is collected, an array of force-distance curves is also collected. This type of data is then called force volume.

Each force curve is measured at a unique X,Y position in the scan area. Force curves from the array of X,Y points are combined into a three-dimensional array, or volume, of force data. The value at a point (X,Y,Z) in the volume is the deflection (force) of the cantilever at that position in space (Figure 6-2).

The force volume data set can be seen as a stack of horizontal slices, each slice representing the array of force data at a given height (as a function of the Z piezo position). A single force volume image represents one of these slices, showing the X,Y distribution of the force data over the scan area at that height. Comparing the topography image to force volume images at various heights can reveal information about the lateral distribution of different surface and/or material properties, including electrostatic, chemical, and magnetic properties.

Similarly, force volume data can be collected with an oscillating cantilever, where the AFM image is typically acquired with TappingMode AFM and the force vs. distance curves plot cantilever amplitude, phase, or even deflection.

#### Advanced Force Spectroscopy

The principles of force spectroscopy can be applied to more advanced kinds of studies using more accurate low-noise AFM instruments (e.g.,  $MultiMode^{\circ}$  PicoForce<sup>TM</sup> or  $NanoMan^{\circ}$  II systems from Veeco Instruments). For example, the relative strengths of the chemical bonds within a single molecule can be measured and compared. The AFM tip can be used to "grab" the end of a molecule, such as a single folded protein. As the cantilever is retracted, the

Figure 6-3. Point-and-click nanolithography.

protein, attached to the tip, unfolds, often one molecular bond at a time. The resulting force-distance curve will show a series of snap-back points in the retraction curve, each representing the breaking of a chemical bond.

#### **Surface Modification Techniques**

Normally, an SPM is used to image a surface without altering it. However, AFMs and STMs can also be used to deliberately modify a surface. This modification is accomplished with specialized SPM software that provides additional ways to control the motion of the scanner and thus the probe or the sample.

#### Nanolithography

Nanolithography is "drawing" a nanometer-scale pattern on a sample surface using an SPM tip. This can be done by mechanically scribing the surface by applying excessive force with an AFM tip or by applying highly localized electric fields to oxidize the surface with an AFM or STM tip. Nanolithography software allows the user to create programs that control tip movements to draw a pre-defined pattern or to draw as freely as when writing by hand with a pen — point-and-click nanolithography (**Figure 6-3**). Nanolithography with an AFM or STM includes recipe-driven modes, or modes in which the nanolithographed pattern is that of a bitmap or vector-based graphic object imported into the SPM software.

#### Nanoindentation, Nanoscratching, Wear Testing

Nanoindentation is a way of measuring mechanical properties by nanoindenting a sample with an AFM tip (often a hard diamond tip) to investigate hardness. Nanoscratching and wear testing utilize the same hardware to drag a tip across the surface with the explicit intent of creating and then visualizing scratches, surface de-laminations, etc. Please see Veeco Instruments' application notes on this subject.

#### Nanomanipulation

Nanomanipulation with AFM requires specialized hardware and software. Advanced software provides the user with flexible control and measurement of in-plane (X,Y) and out-of-plane (Z) position and movement of the SPM probe, using highly accurate positioning hardware and feedback schemes. It allows direct, precise manipulation of nanoscale objects, such as nanotubes and nanoparticles in the plane of the sample surface, and of objects like macromolecules in and out of the plane of the sample surface.



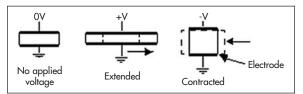


Figure 7-2. The effect of applied voltage on piezoelectric materials.

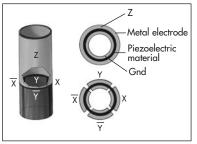


Figure 7-3.
Typical scanner piezo tube and X-Y-Z configurations. AC signals applied to conductive areas of the tube create piezo movement along the three major axes.

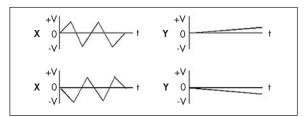


Figure 7-4. Waveforms applied to the piezo electrodes during a raster scan with the X-axis designated as the fast axis (scan  $angle = 0^{\circ}$ ).

A piezoelectric scanner is used in virtually all scanning probe microscopes as an extremely fine positioning element to move the probe over the sample (or the sample under the probe). The SPM electronics drive the scanner in a type of zigzag raster pattern, as shown in Figure 7-1.

While the scanner is moving across a scan line, the data with which the SPM creates the image(s) is sampled at equally spaced intervals.

The in-plane (X,Y) spacing between two consecutive data points in a scan line is called the step size. The step size is determined by the full scan size and the number of data points per line. In a typical SPM, scan sizes run from tens of Ångstroms to over 100 microns and from 64 to 1024 data points per line. The number of lines in an image usually equals the number of data points per line. The image is usually a square; however, rectangular images, as well as images where the number of lines and the number of data points per line are not equal, are also possible.

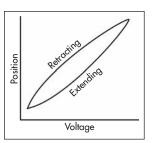
#### **How Scanners Work**

SPM scanners are made from piezoelectric material, which expands and contracts proportionally to an applied voltage. Whether they elongate or contract depends upon the polarity of the voltage applied. All Veeco scanners have AC voltage ranges of +220V- -220V for each scan axis (Figure 7-2).

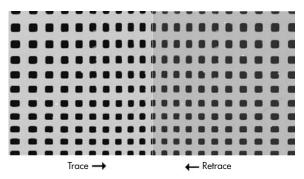
The scanner is constructed by combining independently operated piezo electrodes for X, Y, and Z into a single tube, making a scanner that can manipulate samples and probes with extreme precision in three dimensions.

In some models (e.g., MultiMode SPM), the scanner tube moves the sample relative to the stationary tip. In other models (e.g., STM, Dimension<sup>TM</sup> Series, and BioScope<sup>TM</sup> SPMs) the sample is stationary while the scanner moves the tip (**Figure 7-3**).

AC voltages applied to the different electrodes of the piezoelectric scanner produce a scanning raster motion in X and Y. There are two segments of the piezoelectric crystal for X (X and X) and Y (Y and Y) (Figure 7-4).



**Figure 7-5.** Effect of nonlinearity and hysteresis.



**Figure 7-6.** 100µm x 100µm scans in the forward (trace) and reverse (retrace) directions of a two-dimensional 10µm pitch grating without linearity correction. Both scans are in the down direction. Notice the differences in the spacing, size, and shape of the pits between the bottom and the top of each image. The effect of the hysteresis loop on each scan direction is demonstrated.

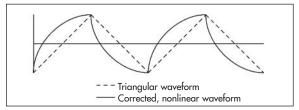


Figure 7-7. Nonlinear waveform (solid line) applied to the piezo electrodes to produce linear scanner movement. The unaltered triangular waveform (dashed line) is included for reference.

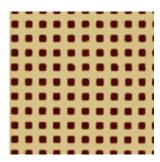


Figure 7-8. 100µm x 100µm scan of the same two-dimensional 10µm pitch calibration grating with a nonlinear scan voltage. Notice the equal spacing between all pits and the consistent shape and size of pits throughout the image.

#### **Hysteresis**

Because of differences in the material properties and dimensions of each piezoelectric element, each scanner responds differently to an applied voltage. This response is conveniently measured in terms of sensitivity, a ratio of piezo movement to piezo voltage (i.e., how far the piezo extends or contracts per applied volt).

Sensitivity does not have a linear relationship with respect to scan size. Because piezo scanners exhibit more sensitivity (i.e., more movement per volt) at the end of a scan line than at the beginning, the relationship of movement vs. applied voltage is nonlinear. This causes the forward and reverse scan directions to behave differently and display hysteresis between the two scan directions.

The effect of nonlinearity and hysteresis can be seen from the curve above (Figure 7-5). As the piezo extends and retracts throughout its full range, it moves less per applied volt at the beginning of the extension than near the end. The same is true when the piezo is retracting: the piezo moves less per applied volt at the beginning than near the end.

Nonlinearity and hysteresis can cause feature distortion in SPM images if not properly corrected. Examples of these effects on AFM images are shown in **Figure 7-6** with a 10µm pitch calibration grating. The depth of each square is 200nm. Please refer to the standard for step height information.

This nonlinear relationship is corrected through a patented calibration routine (Veeco Instruments) by applying a nonlinear voltage in Realtime to produce a linear scan in X and Y in both trace and retrace scan directions (Figure 7-7 and Figure 7-8).

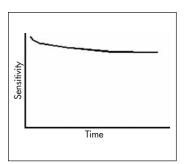


Figure 7-9. Sensitivity vs. operation time.

# Figure 7-10. Image of a calibration grating.

#### **Aging**

The sensitivity of piezoelectric materials decreases exponentially with operation time. Therefore, most of the change in sensitivity occurs at the beginning of a scanner's life, as shown in **Figure 7-9**.

Before Veeco scanners are shipped they run approximately 48 hours in order to get past the point where sensitivity changes dramatically over short periods of time. As the scanner ages, its sensitivity will change less with time, eventually reaching the point where recalibration is seldom needed.

#### Creep

Creep is the drift of the piezo displacement after a DC offset voltage is applied to the piezo. This may occur with large changes in X and Y offsets, and when using the Frame Up and Frame Down commands when the piezo travels over most of the scan area to restart the scan.

When a large offset is performed, the scanner stops scanning and a DC voltage is applied to the scanner to move the requested offset distance. However, the scanner does not move the full offset distance all at once. It initially moves the majority of the offset distance quickly, and then slowly moves over the remainder. The scanning resumes after a majority of the offset distance has been moved, although the scanner is still slowly moving in the direction of the offset.

This slow movement of the piezo over the remainder of the offset distance once scanning has resumed results in creep. Creep appears in the image as an elongation and stretching of features in the direction of the offset for a short period of time after the offset.

An example of creep is shown in Figure 7-10. The tip was scanning from top to bottom and an offset of 10µm in the X direction was performed near the beginning of the scan (indicated by the arrow). The slight bending of the lines that occurs directly after performing the offset is due to creep. The creep settles out by the end of the scan.

When creep appears in an image, it will often settle out by the end of the scan and the next image may be captured. For very large offsets (>50 $\mu$ m), it may take longer than one scan for the creep to settle out. The creep can be reduced by offsetting beyond the desired point and then offsetting back to the desired point.

#### Bow

Because scanners are attached at one end and move the sample or tip on the other, the free end does not move in a level plane. The mechanical properties of the piezo, as well as the kinematics of motion, often result in  $2^{nd}$  order or  $3^{rd}$  order curvatures from an ideal plane. This is commonly called bow, which increases with scan size. The bows can be removed from a captured image by using special software.

#### VIII. Probes

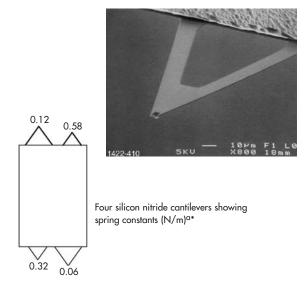
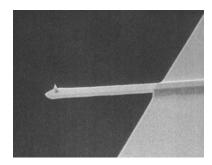


Figure 8-1. Image of silicon nitride probe.



**Figure 8-2.** Scanned electron microscope (SEM) images of silicon cantilever and tip.

#### **AFM Probes**

The next three sections discuss various probe characteristics and types.

#### Silicon Nitride

Silicon nitride probes consist of a cantilever integrated with a sharp tip on the end. The properties and dimensions of the cantilever play an important role in determining the sensitivity and resolution of the AFM.

For contact mode AFM imaging, it is necessary to have a cantilever that is soft enough to be deflected by very small forces (i.e., small force constant) and has a high enough resonant frequency to not be susceptible to vibrational instabilities. This is accomplished by making the cantilever short (to provide a high resonant frequency) and thin (to provide a small force constant).

For the silicon nitride tips, there are four cantilevers with different geometries attached to each substrate, resulting in four different spring constants (Figure 8-1).

Characteristics of the standard silicon nitride probes (Models NP and DNP) are listed in **Table 8-1**.

Spring constant (k)	0.58, 0.32, 0.12, 0.06 N/m*
Nominal tip radius of curvature	20-60nm
Cantilever lengths	100 and 200µm
Cantilever configuration	V-shaped
Reflective coating	Gold
Sidewall angles	35° on all four sides

Table 8-1. Silicon nitride probe characteristics.

#### Silicon

Silicon probes are used primarily for Tapping/Mode applications. The tip and cantilever are an integrated assembly of single-crystal silicon produced by etching techniques. Only one cantilever and tip are integrated with each substrate. These probes (Figure 8-2) can be much stiffer than silicon nitride probes, resulting in larger force constants and resonant frequencies.

Characteristics of the Tapping/Mode Etched Silicon Probe (Model TESP) are listed in Table 8-2.

Spring constant (k)	20-100 N/m
Resonant frequency	200–400 kHz
Nominal tip radius of curvature	5-10nm
Cantilever length	125µm
Cantilever configuration	Single beam
Reflective coating	Uncoated, optional Al coating

Table 8-2. TappingMode Etched Silicon Probe (TESP) characteristics.

<sup>\*</sup> Calculated spring constant values are based on the 0.6µm silicon nitride thickness; however, this value can actually vary from 0.4µm-0.7µm. Thickness is cubed in the spring constant calculation; thus, actual values can vary substantially.

#### **Types of SPM Probes**

#### • Contact Mode AFM Probes

Standard Silicon Nitride Probes (described previously)
Oxide-sharpened Silicon Nitride (Standard) Probes
Oxide-sharpened Silicon Nitride Oriented Twin Tip Probes
Olympus Oxide-sharpened Silicon Nitride Probes
Contact Etched Silicon Probes

#### • TappingMode AFM Probes

TappingMode Etched Silicon Probes (described previously)
Hardened TappingMode Etched Silicon Probes
TappingMode Focused Ion Beam (FIB) Machined Silicon Probes
Olympus Tapping Tips

#### • Probes for Other AFM Techniques

Force Modulation Probes

Force Modulation Etched Silicon Probes

### Magnetic Force Microscopy (MFM) & Electric Force Microscopy (EFM) Probes

Magnetic Force Etched Silicon (Standard) Probes Low Moment Magnetic Force Etched Silicon Probes Low Coercivity Magnetic Force Etched Silicon Probes

#### Scanning Thermal Microscopy

AFM Probes with Integrated Thermal Sensor for Scanning Thermal Microscopy

#### Nanoindentation

Diamond-tipped Cantilever for Nanoindentation/Scratching

#### Probes for Electrical Techniques

PIT - Ptlr-coated Si Probes for SCM and TappingMode
PIC - Ptlr-coated Si Probes for Contact Mode SCM
DDESP - Doped Diamond-coated Si Probes (w/ medium to high spring constant)

#### • Probes for Scanning Tunneling Microscopy (STM) Techniques

Platinum-Iridium STM Tips Tungsten STM Tips

#### IX. Tip Shape Issues

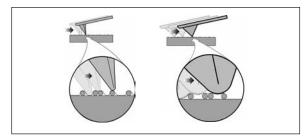
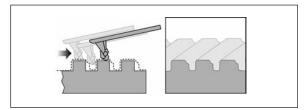


Figure 9-1. Schematics and image profiles of spheres scanned with a sharp (left) and dull (right) probe.



**Figure 9-2.** Schematic and image profile of trenches scanned with a dirty tip.

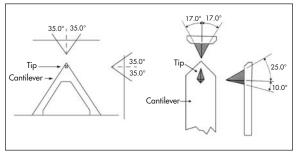
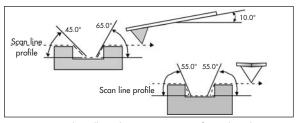
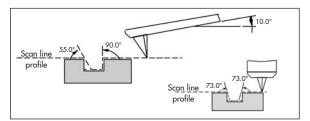


Figure 9-3. Comparison of tip sidewall angles.



**Figure 9-4.** Sidewall angle measurements of trench with vertical sidewalls acquired with silicon nitride probe.



**Figure 9-5.** Sidewall angle measurements of trench with vertical sidewalls acquired with silicon probe.

The SPM image is a result of the interaction of the tip shape with the surface topography. There are two primary features of the tip that affect the SPM image: the radius of curvature and the tip sidewall angles.

The smaller the radius of curvature, the smaller the feature that can be resolved. A sharper tip will be able to laterally resolve smaller features than a dull tip with a larger radius of curvature (Figure 9-1).

The accumulation of debris on the end of the tip can also dull the tip and result in image distortion (Figure 9-2).

**Note:** A dull or dirty tip may not affect the measurement of the vertical dimensions of these samples.

The ability to image steep sidewalls on a sample surface is determined by the sidewall angles of the tip (Figure 9-3); the tip is not able to profile sides of surfaces steeper than the sidewall angle of the tip. When scanning across features that are steeper than the sidewall angle of the tip, the sidewall angle in the images will reflect the sidewall angle of the tip.

The following examples show the resulting angles and image profiles of a silicon nitride tip (Figure 9-4) and a silicon tip (Figure 9-5) scanned over trenches with 90° sidewall angles.

Each tip was mounted in the cantilever at  $\sim 10^{\circ}$ , so the angles measured with the front and back sides of the tip are skewed by  $\sim 10^{\circ}$ .

#### **Resolution Issues**

The resolution of AFM images can be thought of in terms of lateral (X,Y) resolution and vertical (Z) resolution.

#### **Lateral Resolution Issues**

**Tip Shape** — As discussed in the previous section on tip shape effects, the radius of curvature of the end of the tip will determine the highest lateral resolution obtainable with a specific tip. The sidewall angles of the tip will also determine its ability to probe high-aspect-ratio features.

**Pixelization** — The Samples/ line parameter in the Realtime menu determines the number of data points present in the image in the X and Y scan direction. These data points are commonly called pixels. Available choices are 512, 256, and 128.

Pixelization affects resolution in that you cannot resolve features smaller than the pixel size of the image.

For example, if you are acquiring a  $50\mu m \times 50\mu m$  image with the Samples/ line parameter set to 512, then the pixel size is  $98nm (50\mu m \div 512 = 0.098\mu m)$ . Thus, you will not be able to resolve features smaller than 98nm at a  $50\mu m$  scan size.

If you want to be able to see 10nm-sized features, you will need to choose a scan size  $<5.12\mu m$  (preferably 1 or  $2\mu m$ ) since  $5.12\mu m \div 512 = 0.010\mu m$  or 10nm.

Regardless of the pixel size, the feedback loop is sampling the topography many times at each pixel. The data displayed at each pixel is the average of the sampling iterations by the feedback loop over the pixel area.

#### **Vertical Resolution Issues**

**Scanner** — The resolution in the vertical direction is not determined by the tip shape; it is primarily determined by the resolution of the vertical scanner movement, which is  $<1\,\text{Å}$ .

**Pixelization** — As in the lateral dimension, the number of data points in the vertical direction limits the size of the smallest resolvable height change. The number of data points in the vertical dimension is determined by the conversion of 16 bits over the full vertical range of the scanner (440V). The Z limit parameter may be used to increase the sampling resolution in the vertical dimension to image sub-Ångstrom height changes with large Z range scanners.

**Noise** — The overall system noise will generally be the primary limiting factor in the highest obtainable vertical resolution that can be acquired with your SPM. This may be a result of combined effects from electrical, mechanical, and acoustic noise sources. Veeco Instruments guarantees 0.3 to ~1Å RMS noise levels depending on the type of SPM and SPM environment.

#### X. Typical Image Artifacts

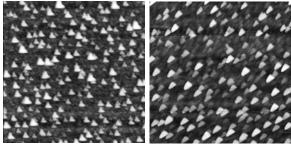


Figure 10-1. Dull or dirty tip.

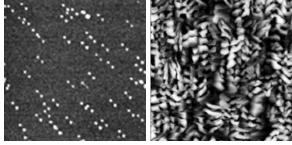


Figure 10-2. Double or multiple tips.

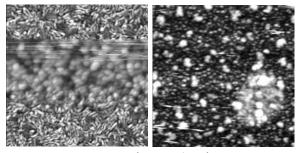


Figure 10-3. Contamination from sample surface.

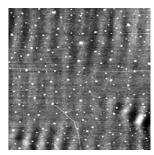


Figure 10-4. Optical interference.

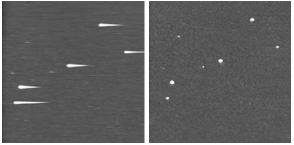


Figure 10-5. Not tracking.

If the tip becomes worn or if debris attaches itself to the end of the tip, the features in the image may all have the same shape. What is really being imaged is the worn shape of the tip or the shape of the debris, not the morphology of the surface features (Figure 10-1).

Double or multiple tip images are formed with a tip with two or more end points that contact the sample while imaging (Figure 10-2).

Loose debris on the sample surface (Figure 10-3) can cause loss of image resolution and produce streaking in the image. The image on the left is an example of loss of resolution due to build up of contamination on the tip when scanning from bottom to top. The small elongated features are represented as larger, rounded features until the debris detaches from the tip near the top of the scan. The image on the right is an example of skips and streaking caused by loose debris on the sample surface. Often, loose debris can be swept out of the image area after a few scans, making it possible to acquire a relatively clean image. Skips can be removed from a captured image with software.

Interference between the incident and reflected light from the sample surface can produce a sinusoidal pattern on the image (Figure 10-4) with a period typically ranging between  $1.5-2.5 \mu m$ . This artifact is most often seen in contact mode on highly reflective surfaces in force calibration plots and occasionally in TappingMode images. It can usually be reduced or eliminated by adjusting the laser alignment so that more light reflects off the back of the cantilever and less light reflects off the sample surface, or by using a cantilever with a more reflective coating (MESP, TESPA).

When the feedback parameters are not adjusted properly, the tip will not trace down the back side of the features in TappingMode (Figure 10-5). The image on the left was scanned from left to right (trace) producing tails on the colloidal gold spheres. The image on the right is the same area imaged with a lower setpoint voltage, increased gains, and slower scan rate.

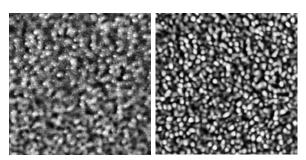
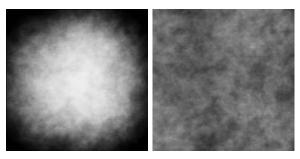
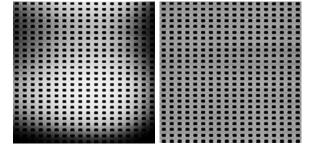


Figure 10-6. Rings during high-frequency operation.



**Figure 10-7.** Large scan size, second order bow. Data scale = 30nm.



**Figure 10-8.** Large scan size, third order bow. Data scale = 324nm.

In TappingMode, when operating on the high-frequency side of the resonance peak (Figure 10-6), rings may appear around raised features that make them look like they are "surrounded by water," as in the image of Ti grains on the left. Decreasing the drive frequency during imaging can eliminate this artifact, as shown in the image on the right. (When reducing the drive frequency, the setpoint voltage may need to be reduced, too.)

The arch-shaped bow in the scanner becomes visible at large scan sizes (Figure 10-7, left). The bow in this image can be removed by performing a second order planefit in X and Y (Figure 10-7, right).

At large scan sizes, the bow in the scanner may take on an S-shaped appearance (Figure 10-8, left). The bow in this image can be removed by performing a third order planefit in X and Y (Figure 10-8, right).



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