



AFM Almanac

Imaging Modes

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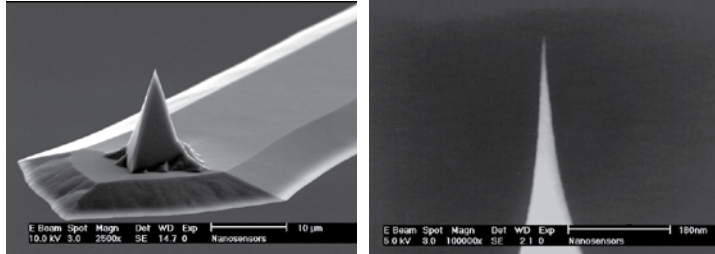


Scanning Probe Microscopy (SPM)

Scanning Probe Microscopy (SPM) is a large and growing collection of instrumentation methods for investigating the properties of a sample at or near the sample surface. The SPM instrument has a sharp probe (radius of curvature typically in the nanometers or 10's of nanometers) that is in near-contact, or perpetual contact, or intermittent contact with the sample surface.

When we say an SPM is used to investigate sample properties at or near the sample surface, by near the surface, we mean immediately beneath the surface (typically several nanometers deep) and immediately above the surface (typically up to several tens of nanometers high). This is a limitation in the sense that the bulk of a thick specimen is generally inaccessible to SPM methods. Nonetheless, today there are tens and possibly over a hundred different SPM techniques, and the list of their applications in science and engineering is growing.

AFM Cantilever and Tip



SPMs were first used to create 3-dimensional images of the sample surface, and even though that is still the main application, other techniques that do not always involve 3-dimensional images have ensued and the sphere of methodology and applications keeps expanding. In this tutorial, we will visit some of the techniques and their applications.

The small, finite size of the probe's sharp end is one key element that gives the SPM its resolution power, but it is also a limitation of SPM. As the probe can only interact with a small area of the sample surface at any given time, it becomes necessary to raster-scan either the sample or the probe in the XY plane to cover an area and create an image of that area.

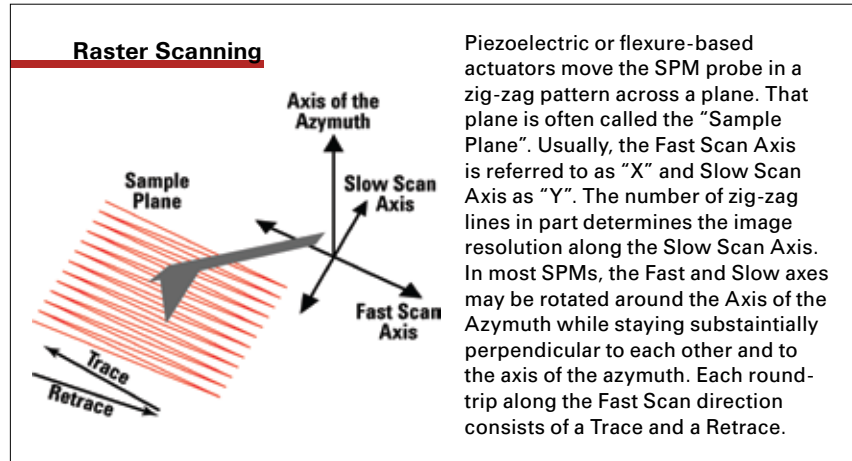
Raster Scanning

SPM relies on raster scanning to probe an area of the sample surface. Raster scanning is performed with actuators whose motion can be incremented in small steps and with high precision. These actuators are usually made of piezoelectric materials shaped into the form of a hollow tube, or they are made of mechanical flexures, or a hybrid of the two.

Raster scanning makes it possible to record the probe-sample interaction point-by-point. For each X,Y coordinate pair, the interaction is recorded as one data point. The collection of these data points is then synthesized into the SPM image, a 3-dimensional map. In other words, the area of the sample surface under study is probed not all at once, but in a time series of measurements that are

put together point-by-point, and line-by-line. This is why the instruments are called Scanning Probe Microscopes. In this sense, SPMs are identical to scanning electron microscopes (SEM), except that instead of an electron beam, SPMs use mechanical, opto-mechanical, or electrical probes to interact with and to interrogate the sample surface.

Raster scanning requirement is a major speed bottleneck in SPM. The scanning speeds in SPM have traditionally been slower than in SEM, but this gap is closing fast, and is even projected to be reversing in the near future.



SPM Images

Although the word "map" describes them more accurately, SPM maps are most often called "images," and in keeping with the common practice, we will usually use the word image.

The most common SPM images are topography images, which are most often created with Atomic Force Microscopy (AFM) or Scanning Tunneling Microscopy (STM). We will study both AFM and STM in some detail. The acronyms AFM and STM also stand for atomic force microscope, which was invented in 1986,¹ and its predecessor scanning tunneling microscope, invented in 1982.²

In AFM topography images, the third dimension, Z, at a given X,Y coordinate pair, is the relative height of the sample surface at those coordinates. This interpretation implies the AFM's sharp probe does not deform the sample surface, either reversibly or irreversibly. The harder the sample surface, the more accurate is this interpretation, because the AFM tip penetrates a harder sample surface less than it does a softer one. In other words,

the AFM tip follows the height variations of hard surface with higher fidelity than it does soft surfaces.

AFM height measurements are in general calibrated against height standards. Those height standards themselves are often measured with methods other than SPM. In this sense, AFM topography (height) images may often readily be compared for quantitative information, even if they are collected on different instruments, so long as the calibrations are accurate; the instruments work well; and the AFM users operate the instrument properly, and know how to correctly extract information from the raw data.

In STM topography images, the interpretation of the third dimension, Z, as the relative height of the sample surface is sometimes less straight forward than it is in AFM. (See the section on Scanning Tunneling Microscopy).

In other types of SPM images, the third dimension of the image is a measure of the

relative strength of a detectable interaction, between the probe and the sample, that may or may not have a correlation with the topography. The image is usually recorded simultaneously with, and displayed side-by-side, the topography image of the same in-plane coordinates (same area on the sample). This helps reveal any correlation that may exist between topography and the interaction recorded in the second image.

In a few cases, the measurement of the interaction may be calibrated, so that in addition to mapping the relative strength of this interaction, the image may also yield quantitative information beyond topography about the sample. An example is Surface Potential image or Scanning Kelvin Probe Microscopy (SKPM) image. But generally, non-topographic SPM images serve to identify only relative variations of the interaction.

¹ G. Binnig, C. Gerber, C. Quate, *Physical Review Letters*, 56, p 930, 1986.

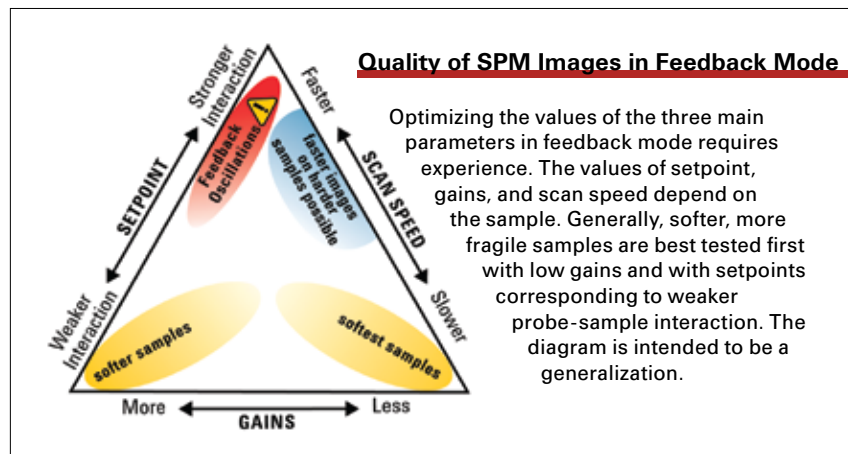
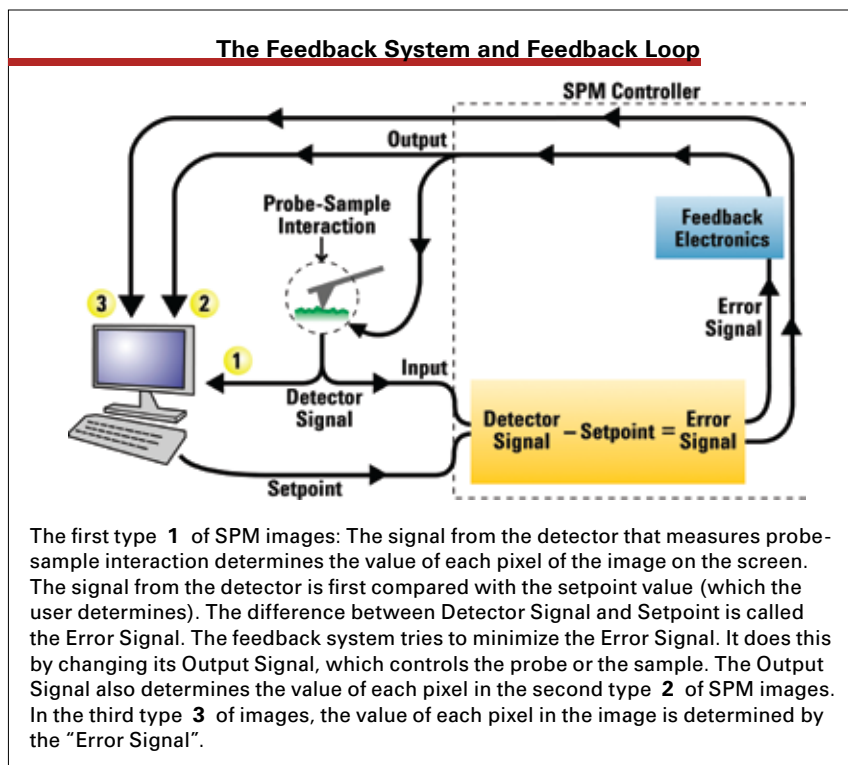
² G. Binnig, H. Rohrer, C. Gerber, E. Weibel, "Surface studies by scanning tunneling microscopy," *Physical Review Letters*, 49, pp. 57-61, 1982.

A Classification of SPM Image Types

Generally, SPM images can be categorized into two groups. In the first group, the quantity that is mapped pixel by pixel at each X,Y coordinate to construct the image, is a measure of the value of the signal that comes from a detector, without any feedback on that signal. This signal measures the strength (and sometimes also records the polarity) of the interaction between the probe and the sample surface. It may be, for example, the deflection of the microfabricated cantilever in the AFM, or the current through the metal tip in the STM.

In the second group, the signal coming from the detector serves as the input of a feedback system, the output of which is mapped at each X,Y coordinate to construct the image. The feedback system strives to maintain the value of the detector signal at a user-defined setpoint value. Operating any SPM properly in a feedback mode requires a solid understanding of the fundamental concepts of a feedback system. This does not require an engineering background; the concepts are intuitively simple to grasp. However, becoming comfortably familiar with them requires some practice with the instrument, which is best done using a well-characterized sample, for example, a relatively inexpensive reference sample used for calibrating the scanner.

The three most important parameters to change in optimizing the quality of an SPM image in a feedback mode are setpoint, gains, and scan speed (or scan rate). Faster scan speeds require more aggressive feedback, which means higher feedback gains. There is a limit to how much the gains can be



increased; too much gain will result in the problem of feedback oscillations which shows up in the image. The value of the setpoint reflects the desired strength of the signal at the detector, which in turn is related to the strength of the tip-sample interaction.

When there is no feedback, the strength of the probe-sample interaction may vary significantly

across the raster-scanned area. For example, if an AFM is operating with no feedback on sample topography, then the tip-sample force will change drastically with large variations of topography, and this may lead to irreversible changes of the sample and the tip. For this reason, AFM topography images are nearly always collected in feedback mode. (See Contact Mode AFM).

A Classification of SPM Image Techniques

In general, we can categorize Scanning Probe Microscopy (SPM) techniques into three groups: Imaging Techniques, Non-imaging Techniques, and Hybrid techniques which combine the latter two.

Imaging Techniques

Imaging Techniques can be subdivided into two groups of "Modes" we call Primary Imaging Modes, and Derivative Imaging Modes. This classification is arbitrary, some may argue, but it has shown to serve a useful pedagogic purpose by helping us manage our knowledge of the increasingly long and complex list of SPM techniques that continue to be invented.

At this time, we can identify at least five types of Primary Imaging Modes widely in use:

- Scanning Tunneling Microscopy (STM)
- Quasi-static or Contact Mode Atomic Force Microscopy (AFM)
- Dynamic Vertical Mode(s) of Atomic Force Microscopy
- Dynamic Lateral Mode Atomic Force Microscopy
- Scanning Near-field Optical Microscopy (NSOM).

The main features that distinguish these five modes each from each are the nature of the probe tip-sample interaction (electrical, mechanical, optical, or a combination of these), the time-scales involved in the interaction, and the proximity of the sample to the probe tip. The time scales are determined in part by the presence or absence of probe tip oscillations, and these oscillations also affect tip-sample proximity or contact.

For example, STM relies primarily on the electrical interaction between the probe tip and the sample, AFM on the mechanical. Contact Mode AFM differs from Dynamic Vertical Modes of AFM in the duration of probe tip-sample interaction. Dynamic Lateral Mode is also different from Dynamic Vertical Modes. NSOM relies primarily on a combination of optical and mechanical interactions between the probe tip and the sample.

Each of these Primary Imaging Modes enables a host of Derivative Imaging Modes. For example, Dynamic Vertical Modes and Dynamic Lateral Mode have a derivative mode

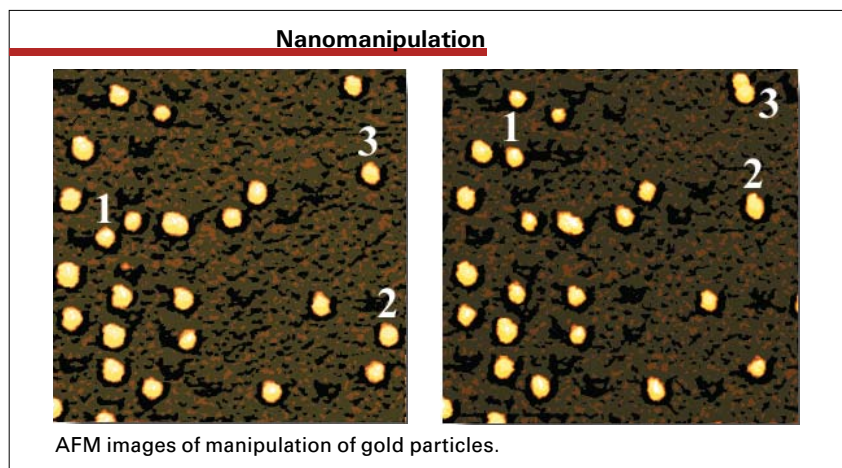
called Phase Imaging which has proven very useful in material research: visco-elasticity, adhesion, electrical, and magnetic properties. Contact Mode AFM has a derivative mode called Lateral Force Microscopy (LFM) which has significantly advanced our understanding of friction and tribology on the nanometer scale.

Non-Imaging Techniques

In addition to imaging techniques (and their classification into primary and derivative modes), we can identify a very broad, second class of techniques, which is related to the imaging techniques in the sense that they rely on the same tip-sample interactions as do the Imaging Techniques, but which extend the utility and applications of the SPM beyond imaging.

Collectively, these can be called Non-Imaging Techniques. Two closely related examples of non-imaging techniques are nano-indenting and nano-scratching with applications in mechanical and wear testing of materials. Here, the AFM tip is used to image the sample surface, then deliberately indent it or scratch it, and then image the altered area to visualize the change. For indenting and scratching hard surfaces, the tip is often made of a very hard material such as diamond-like-carbon-coated silicon, or even a single micro-crystal of diamond.

Nano-indenting and nano-scratching are examples of a broader class of non-imaging techniques that are generally referred to as nano-manipulation. In some nano-manipulation techniques, we can modify



the sample surface with more control and finesse than in nano-indenting and nano-scratching. For example, we can use the AFM or the STM tip to rearrange nanometer-scale objects physisorbed on that surface. Essentially, the tip serves as a nano-scale finger to interact with the sample.

Nano-manipulation is sometimes performed in the plane of the sample surface (in-plane) and sometimes out of this plane (out-of-plane nano-manipulation). An example of out-of-plane nano-manipulation is attaching an AFM tip to the end of a macromolecule on the sample surface, and pulling the molecule so that its secondary or tertiary structure unfolds. This is now an extremely

active area of research, with applications extending to areas as diverse as drug discovery and composite materials design.

In Non-Imaging techniques, we can group together several that have much in common, and refer to them as Spectroscopy Modes, or Scanning Probe Spectroscopy. Spectroscopy modes are closely related to their respective modes in the Imaging Techniques as we will discuss elsewhere in this tutorial. In Spectroscopy Modes, raster scanning in an imaging mode is disabled to allow the SPM to record the interaction between the sample and the probe tip at a given point in the sample plane (a given X,Y coordinate), while we change one or more parameters, by

either stepping them in discrete quanta, or by ramping them at a rate we control.

Scanning Probe Spectroscopy was recognized early on as a useful technique, but in light of intense interest in protein interactions and research into synthetic and naturally-occurring macromolecules, it has become widely used and highly in demand since the late 1990's.

Hybrid Techniques

An example of a Hybrid Technique is Force Volume Mode, which combines Force Spectroscopy and Imaging, either in Contact Mode AFM, or in Intermittent Contact Mode AFM.

Contact Mode or Quasi-static Atomic Force Microscopy (AFM)

In contact mode AFM, the sharp tip at the end of the micro-fabricated AFM cantilever is in perpetual contact with the sample surface. This can be done with the cantilever bent up or bent down from its free equilibrium position, the latter due only to attractive forces between the tip and the sample. The spring constant k (typically 0.001 - 5 nN/nm for Contact Mode) of the AFM cantilever and the deflection z (typically few nm, up to several 10's of nm's for Contact Mode) of the cantilever from its free equilibrium position define the minimum value of the force F_c between the tip and the surface in contact mode AFM when the deflection is up:

$$F_c^{\min} = k \cdot z_{up}$$

The total force is larger than this minimum, because upon

contact between the tip and the sample surface, attractive forces tend to hold the tip and the sample together even if the cantilever were pulled away from the sample so that the cantilever bends down past its equilibrium position.

These attractive forces include capillary and adhesive forces, as well as Van der Waals forces. They result in part from a thin layer of molecules adsorbed on the sample and the tip. The composition of this layer depends on the environmental conditions of the laboratory and also on the tip and the sample material. Hydrocarbons and water molecules are major contributors to this layer in ambient air and also in low to intermediate vacuum. After bringing the tip and the sample into contact, one

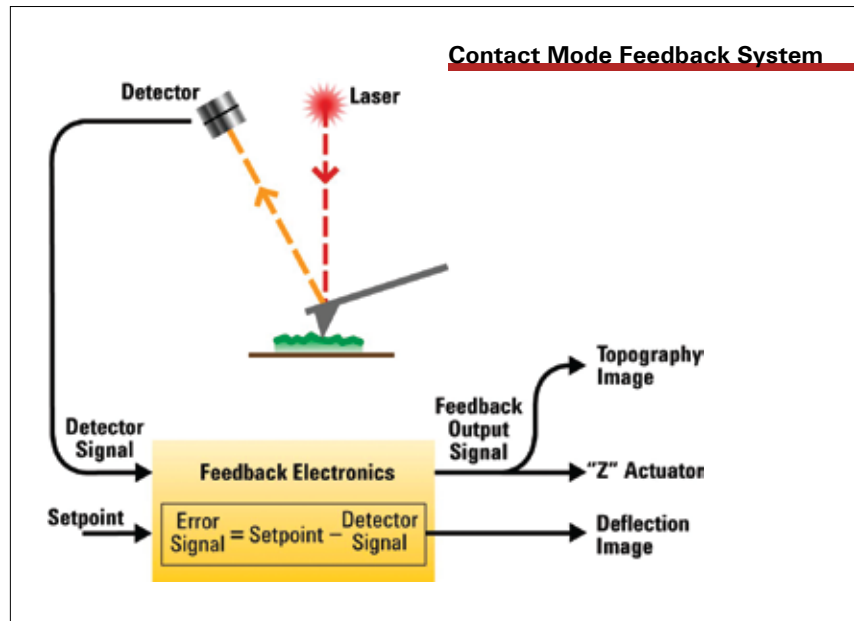
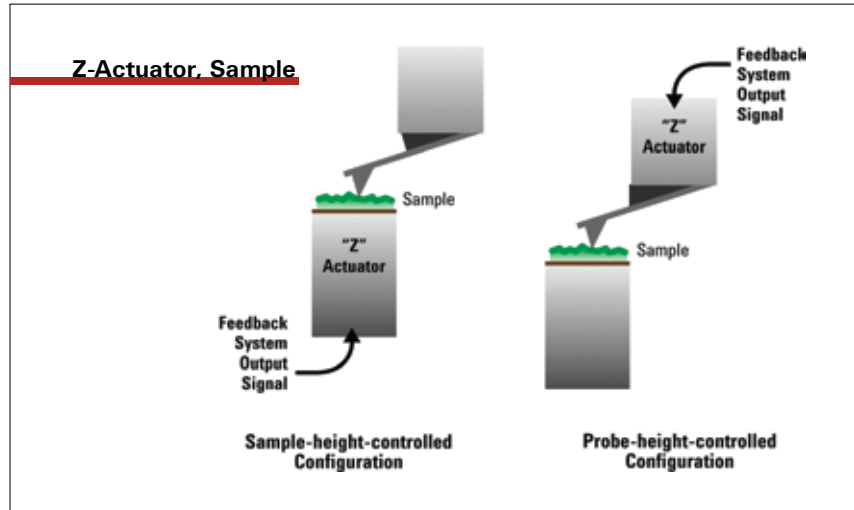
can retract the two and record the deflection of the cantilever down, below its free equilibrium position, in order to get an estimate of the attractive forces.

The signal from the detector is the almost-vertical deflection of the AFM cantilever (deflection signal), measured usually at or near the free end of the cantilever using a reflected laser beam. This signal subtracted from the AFM feedback system's setpoint value is what we call the error signal and is usually recorded in what is known as the Deflection Image - clearly a confusing misnomer, which has unfortunately taken root in the SPM jargon. If there is no feedback on this error signal, then, with proper calibration, and for small cantilever deflections, typically under 500nm, the deflection image can serve as a

topography image on samples with hard surfaces (so that the AFM tip does not significantly indent into the surface).

Usually, the error signal is the input to a feedback system, the output of which (after significant amplification) serves to drive the AFM's Z-actuator, which moves either the sample or the probe in the Z direction. Then, this feedback output signal, which is almost universally called the Z-signal, is the one that is mapped to create the Topography Image.

In this case, it is the Z-actuator whose movement is calibrated. The Z-actuator is typically a piezoelectric tube, or a flexure, or a hybrid of the two. Often, the Deflection Image and the Z-actuator Topography Image are collected simultaneously, with the Deflection Image highlighting the edges of features in the topography image. The Deflection Image on a softer sample often reveals sub-surface structure more clearly than the Topography Image. If the feedback is optimized, then the deflection image is the first derivative of the topography image.



Lateral Force Microscopy (LFM)

LFM is a Derivative mode of Contact Mode AFM. The tip is constantly in contact with the sample surface. In LFM, the fast direction of the raster scanning is perpendicular to the AFM cantilever's long axis, and the cantilever twists about this same axis as the raster scanning proceeds. As a result, in addition to the near-vertical deflection

signal which is usually present during contact mode AFM, the detector can also collect a sizeable lateral deflection signal from the cantilever's twisting motion.

The strength of the lateral deflection signal is related to the friction force between the sample surface and the tip. This is why

LFM is sometimes called Friction Force Microscopy.

The LFM signal is highly susceptible to topography variations; the rougher the sample surface, the more topography convolution. To take out this convolution, to decipher the variation in friction force from the topography, two LFM

images are often captured side by side. One of these two images is constructed from the detector signal during the trace of each round-trip cycle (of the raster scan). The other, during retrace. Then one of the two images is inverted and subtracted from

the other. This reduces the topographic artifacts in the LFM signal.

The LFM signal is one of the those that so far has rarely been used in feedback.

Scanning Tunneling Microscopy (STM)

The Scanning Tunneling Microscope (STM) works well only with (electrically) conducting and semi conducting samples. The STM, a Primary Imaging Mode in SPM, was the first modern SPM, invented in 1982, winning the Nobel Prize in Physics for the inventors. The papers in which the STM invention and its first applications were revealed are the most widely cited papers in subsequent SPM-related publications.³ Despite the limitation that it does not work with electrically insulating samples, the STM has provided the highest resolution images in three dimensions on more samples than any other SPM method.

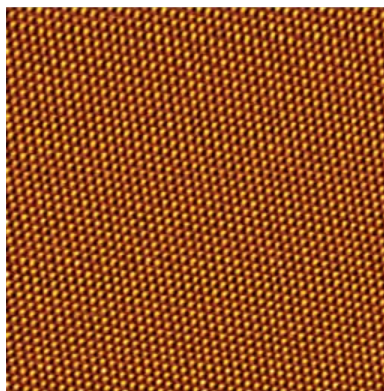
In STM, the probe is a sharp metal tip, typically made of either tungsten, or a platinum-iridium (Pt-Ir) alloy. The detector signal is the tunneling current I between the sample and the tip (typically 100's pA to several nA) when a bias voltage V (typically 10's or 100's mV) is applied between the two. The magnitude of this current is extremely sensitive to, varying exponentially with, the small gap δ that separates the nearest atoms between the tip and the sample surface.

$$I \propto V e^{-\kappa\delta}$$

This sensitivity, together with sub-Angstrom positioning precision and control of the instrument's Z-actuator render the STM the highest-resolution SPM in all 3 dimensions. Atomic-scale resolution in STM images, in X, Y, and in Z, are routinely possible on atomically smooth surfaces of metals and semiconductors if the instrument's noise floor is low enough.

When the STM is operated in feedback mode, the tunneling current is the input signal for the feedback system. The output signal usually drives the Z-actuator, (same as in Contact mode AFM). In general, though, this type of image is not always a straightforward representation of the atomic scale topography

STM Atomic Resolution



5500 STM image of HOPG showing atomic structure. Scan size 100 nm

because, the energy sates into and out of which the tunneling current flows across the tip-sample separation depend on numerous parameters, including the strength and the polarity of the bias voltage.

STM's reliance on the electric current limits its usefulness to samples that can sustain a large enough tunneling current that can stand out of the noise signal in the detector electronics. Although low-current STM has extended the use of STM to less conducting samples, in general insulators cannot be imaged with STM, or even with the more sensitive low-current STM.

³ G. Binnig, H.Rohrer, C. Gerber, E. Weibel, "Surface studies by scanning tunneling microscopy," *Physical Review Letters*, 49, pp. 57-61, 1982.

Dynamic Vertical Mode Force Microscopy

In Dynamic Vertical Mode Force Microscopy, the AFM cantilever oscillates, typically at frequencies in the kHz up to 100s of kHz. The oscillations are such that the free end of the cantilever and the tip move along a gently curved trajectory on a plane perpendicular to the XY plane. The tip motion is therefore not strictly perpendicular to the XY plane, but the departure from perpendicularity is often negligible.

The cantilever is usually mounted at an angle relative to the X,Y plane. This angle, which is typically around 10-15 degrees, is necessary to ensure the cantilever's free end and the tip are closer to the sample surface than the cantilever's fixed end, and so that the tip apex is the first point of contact between the sample and the cantilever when the two approach. For those applications that require tip oscillations substantially perpendicular to the X,Y plane, some tips are manufactured in a way that the apex of the tip is angled to compensate for the cantilever's tilt.

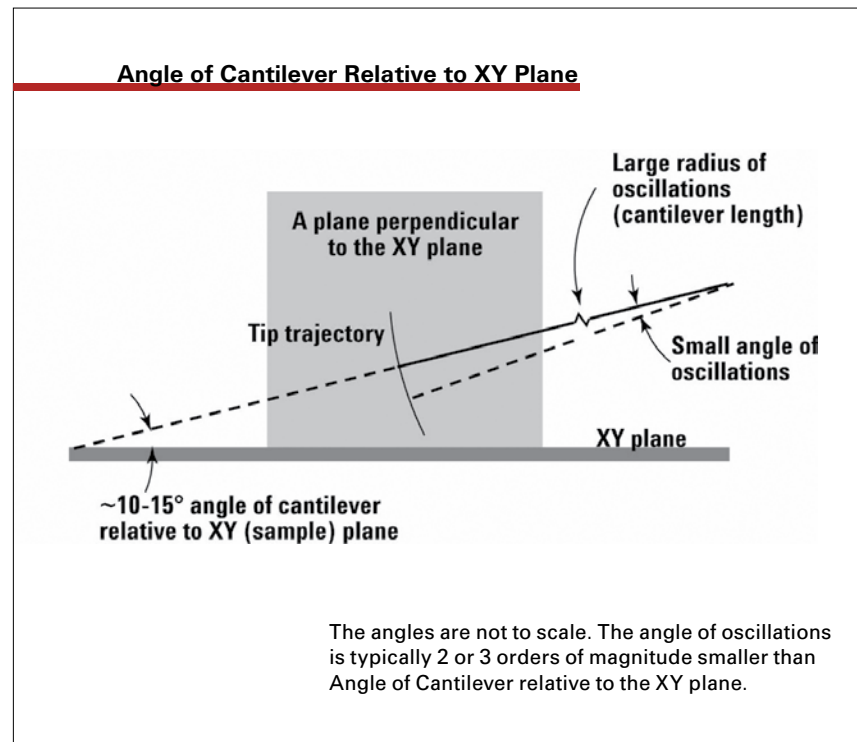
Generally, the amplitude of the oscillations at the free end of the cantilever are in the nanometers or tens of nanometers, and occasionally a few hundred nanometers.

There are at least three methods of operating an AFM in a Dynamic Vertical Mode. These are usually called Non-contact Mode AFM, Frequency Modulation AFM (FM-AFM), and Amplitude Modulation AFM (AM-AFM). The latter is also referred to as Slope Detection Mode AFM. Amplitude Modulation is an unfortunate choice of name, because unlike in FM-AFM, where the instrument modulates the frequency of cantilever oscillations, in AM-AFM, the instrument does not modulate the amplitude. Similarly, Slope Detection is poor nomenclature because the quantity that is detected (and controlled) in this mode is simply the amplitude of the cantilever oscillations, not the

rate of change of one thing with another as the word slope would suggest. But this nomenclature is now widely in use. Here, we use "Intermittent Contact Mode AFM" instead, which is also widely used and adopted in the literature, but which is more accurately descriptive.

Frequency Modulation and Non-contact Mode AFM are described elsewhere in detail, and we do not cover in this tutorial.⁴

⁴ For an excellent review, see "Dynamic atomic force microscopy" by R. Garcia and R. Perez, in *Surface Science Reports*, 2002, 47.



Intermittent Contact Mode AFM

In Intermittent Contact Mode AFM, the cantilever's oscillation amplitude (and phase relative to the drive signal) are the main quantities that are measured. Usually, the quantity that the feedback system strives to control is the cantilever's oscillation amplitude.

The measured amplitude is compared with a setpoint value. The difference between the two is called the error signal, and is the input into the feedback system, the output of which (after amplification) drives the Z-actuator. This output signal is the quantity that is most often plotted for each X,Y coordinate to synthesize the 3-dimensional image that is usually called the Topography Image or the Height Image in Intermittent Contact Mode AFM.

The measured amplitude itself is rarely used to compose an image, but the error signal, the difference between it and the set point value, frequently is. This image is usually displayed and captured side-by-side the topography image, and is called "the Amplitude Image," clearly a confusing choice of name. In many ways the Amplitude Image in Intermittent Contact Mode AFM is similar to the Deflection Image in Contact Mode AFM. On harder samples, the Amplitude Image highlights the edges

of features, and on softer samples, it often depicts sub-surface features better than the topography image does.

Different methods are used to drive the cantilever in all Dynamic Mode AFMs, including in Dynamic Vertical Mode AFM, and specifically, in Intermittent Contact Mode AFM. These methods are described in MAC Mode, and AC Mode.

Regardless of the method of drive, the driving force F is usually sinusoidal:

$$F = F_o \cos(\omega t),$$

$$\omega = 2\pi f$$

Where the drive frequency, f , is typically at or near one of the cantilever's eigenfrequencies. Most often, this is the fundamental eigenfrequency f_o , and the free end of the cantilever oscillates with an amplitude, A , that is proportional to the quality factor Q , of the resonance around the eigenfrequency, and inversely proportional to the cantilever's spring constant k .

$$A = F_o \frac{Q}{k}$$

Absent any tip-sample interactions, the cantilever oscillations, $z(t)$, are also sinusoidal if the drive amplitude, F_o , is small enough to keep the cantilever motion small compared with the cantilever thickness.

$$z(t) = A \cos(\omega t + \varphi)$$

When the cantilever and the sample are close enough, during each oscillation cycle, the tip moves through an interaction potential that includes long-range attractive and short term repulsive components. There are also non-conservative forces that are not accounted for in this potential. The tip-sample forces are highly nonlinear and complex, especially around the lower excursion point of the cantilever, when the tip is closest to the sample or temporarily in contact with it.

One of the main advantages of Intermittent Contact Mode AFM over Contact Mode AFM is that since the tip only intermittently contacts the sample surface, lateral shear forces that may alter (dull) the tip or rearrange (damage) the sample surface during raster scanning are significantly reduced or nearly eliminated. This was the main impetus behind the development of Dynamic Vertical Mode Force Microscopy. But it was soon realized that Dynamic Force Microscopy provides additional cantilever signals to probe the tip-sample interaction, chief among them are the relative phase φ and frequency shift Δf of the cantilever.

Methods for Driving the AFM Cantilever for Intermittent Contact AFM

MAC Mode

Magnetically Actuated AFM (MAC Mode) is an implementation of Intermittent Contact Mode AFM. In MAC Mode, the AFM cantilever is partially coated with a magnetic thin film. An alternating current is driven through a coil that is positioned over the cantilever and may be driven into (near-) vertical oscillations via the inductively-induced magnetic field interacting with the magnetic coating.

In MAC Mode, the drive force is applied directly to the cantilever, close to the cantilever's free end. This feature of MAC Mode makes it substantially more

suitable than Acoustic Mode for Intermittent Contact Mode AFM imaging and Spectroscopy in liquid. The difference between the resonance response of an AFM cantilever in a liquid as driven with MAC Mode and with Acoustic Mode reveals this quite clearly.

AC Mode

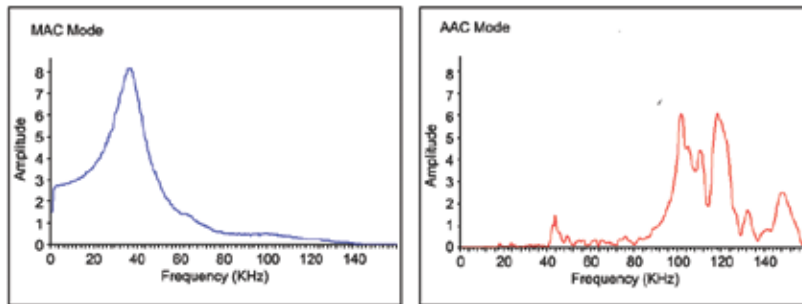
Alternating Current (AC) Mode AFM is another implementation of Intermittent Contact Mode AFM. In AC Mode AFM, the AFM cantilever is driven at its fixed end with a piezoelectric actuator that is positioned under the substrate to which the cantilever is attached. An alternating current (AC) voltage is applied

to this actuator, whose resulting motion is then amplified at the cantilever's free end, where the tip is.

Acoustic Mode

This mode is yet another implementation of Intermittent Contact Mode AFM, used to drive the cantilever in a liquid. The liquid cell is fixed on the sample plate and a piezoelectric actuator drives the nose cone with its cantilever. The cantilever's frequency response is a convolution of its own resonance response with the response of the liquid and the liquid cell. The mass of the cantilever is negligible compared with that of the liquid in motion. The resonance response of the cantilever is overshadowed by other peaks in the frequency response curve in Acoustic Mode. Phase Images in liquid using Acoustic Mode drive are hardly ever as useful as their counterparts in air, or as in liquid with MAC Mode. This lack of clarity in the apparent resonance response also makes the interpretation of the recorded images subject to more uncertainty than images taken with MAC Mode.

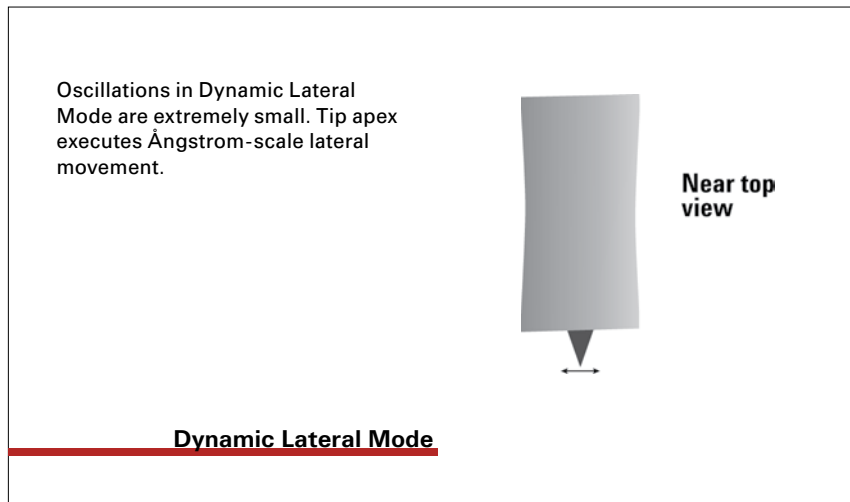
Resonance Curves in Liquid



Dynamic Lateral Mode AFM

In Dynamic Lateral Mode AFM, the AFM cantilever is mechanically driven such that it moves laterally along its long axis. The tip then executes a rigid-back and forth movement on the same axis, in a plane that is perpendicular to the axis.

A unique feature of Dynamic Lateral Mode AFM is that it combines very high-frequency tip motion with extreme tip-sample proximity during the entire oscillation cycle of the cantilever. This is in contrast to Dynamic Vertical Modes of AFM, such as Intermittent Contact AFM and FM-AFM, in which the tip typically spends most of every oscillation cycle relatively far, up to several tens of nanometers, away from the sample surface. Therefore, in Dynamic Lateral Mode AFM, the average quantities measured by the instrument, such as cantilever



amplitude and phase, are much more closely related to tip-sample interaction than they are in Dynamic Vertical Modes.

As in LFM, Dynamic Lateral Mode AFM provides information about lateral interactions between the tip and the sample when

the two are close enough to engage. Because of the lateral oscillations, here unlike in LFM, there is usually no need to capture two images, one of trace and one of retrace, for each cycle of the raster scanning.

Phase Imaging

Phase Imaging is a derivative mode of Dynamic Mode AFM (both Lateral and Vertical) where the phase of the cantilever oscillations ϕ , measured relative to the drive signal oscillations, has turned out to be a powerful signal for studying the properties of the sample surface, especially heterogeneous surfaces. Phase Imaging has become especially useful for polymer research, and for electrical and magnetic property investigations as in Electric Force Microscopy (EFM) and Magnetic Force Microscopy (MFM).

Phase Images often compliment topography images by mapping the various regions of the sample surface each of which interact with the tip in a slightly (or significantly) different way from each. This difference is sometimes so subtle that it is barely noticeable in the Topography Image, but clearly visible in the contrast variations in the Phase Image. More often than not, however, topographic features convolve into the Phase Image, and must be recognized apart from the contrast in the Phase Image that is primarily a result of material inhomogeneity.

Experienced users of Phase Imaging are better able to tell the difference between phase contrast due to differences in material properties from that due to topography convolution.

The origin and the interpretation of the phase signal has been the subject of intense research and debate since the early 1990s, but even so, the utility of Phase Imaging has expanded the applications of Dynamic Modes of AFM into numerous broad areas of research including magnetic and electric properties.

Magnetic Force Microscopy (MFM)

Magnetic Force Microscopy is a derivative mode of Contact Mode AFM and Intermittent Contact AFM. In MFM, the AFM tip is coated with a thin magnetic film, for example, cobalt-chrome (Co-Cr). The magnetic (dipole) moment $\vec{\mu}$ of the AFM tip couples to the stray magnetic field \vec{B} emanating from the sample surface, giving rise to the magnetic force F_m between the tip and the sample. This force, typically in the 10's of pN's, is much smaller than the forces during Intermittent Contact AFM used for topography imaging. To detect this magnetic interaction with MFM as a Derivative of the Quasi-static (Contact) Mode AFM, cantilevers with low stiffness must be used: spring constant typically 0.01-0.1nN/nm. Here, the sample surface is usually very smooth, so that the tip can fly over it at a prescribed height without contacting the

sample, and recording only long-range (magnetic) interactions.

In MFM as a Derivative of a Dynamic Mode (Lateral or Vertical), however, the sensitivity of the cantilever oscillations to small forces make it possible to use much stiffer cantilevers, with spring constant typically in the 10s of nN/nm. The interaction between the tip and the sample's stray magnetic field leads to changes in the amplitude and phase of the cantilever oscillations and also to a change of the resonance frequency of the cantilever.

Since the tip oscillates with a non-zero amplitude, the distance between the tip and the sample changes during each oscillation cycle, and with it so does the strength of the stray magnetic field and the tip-sample magnetic interaction: The gradient of the

magnetic field above the sample surface leads to a gradient of the tip-sample force, which then alters the cantilever's motion. It is this force gradient that the MFM mode detects and maps into the image.

At a given drive frequency f , the phase φ and the amplitude A of the cantilever oscillations change. These changes can be recorded pixel by pixel to generate an MFM image. When the phase signal is mapped in this way, an MFM Phase Image is created. Alternatively, the phase signal can be used in a feedback loop, which controls the frequency of the drive signal in such a way to maintain a constant phase difference between the drive signal and the cantilever oscillations. In this case, one obtains a frequency modulation MFM image (FM-MFM).

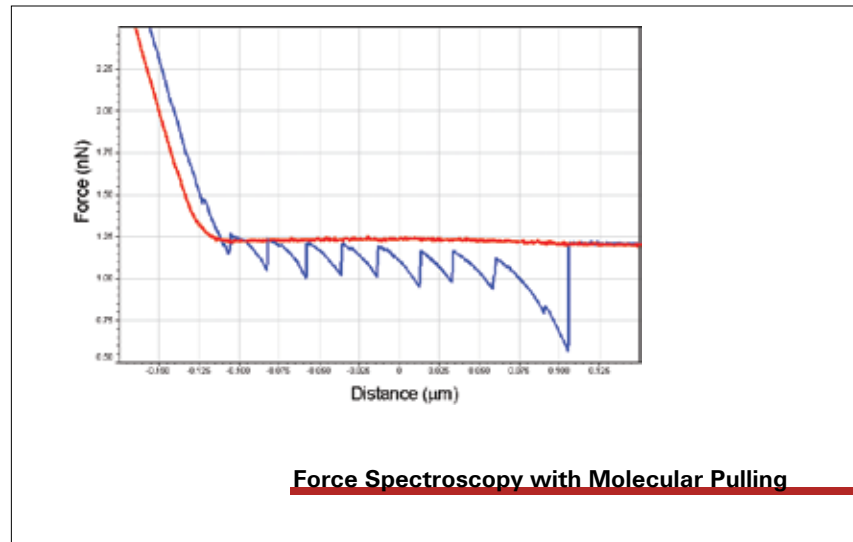
SPM Spectroscopy

Traditionally, spectroscopy refers to the study of the emission or absorption of energy. The spectrum of values at which an object absorbs or emits energy provide information about the atomic and molecular composition and structure of the object. In electromagnetic radiation spectroscopy, the energy is proportional to the frequency of the radiation, so that spectroscopy becomes a study of emission or absorption of radiation as a function of the radiation frequency (or wavelength).

One of the main features of an SPM is its ability to make highly localized measurements of the interaction between the sample surface and the probe. In Imaging Modes, this feature is used together with precision raster scanning in X and Y, to generate various types of high resolution images of the sample surface, e.g., topography (or height) image. If raster scanning is disabled, then the interaction between the sample and tip can be studied at a given X,Y coordinate, and this takes us to the subject of Scanning Probe Spectroscopy.

NSOM lends itself very well to spectroscopy in the traditional sense, but with a much enhanced lateral (X,Y) resolution. Highly-localized optical spectroscopy, including with sub-wavelength resolution, constitute a major application area for NSOM. This is called Near-field Optical Spectroscopy

Scanning Tunneling Spectroscopy (STS) was the first spectroscopy technique that used an SPM. Here, the energy levels



of an object (e.g., a molecule or an atom) into and out of which a tunneling current flows are studied, as the bias voltage across the tip and the sample changes--in magnitude and in polarity. To the extent that STS probes energy levels directly, it also fits the traditional definition of spectroscopy.

With the invention of the AFM, however, the definition of spectroscopy was expanded to include not only energy, but also force. In AFM Force Spectroscopy, the raster scanning is stopped, allowing for the AFM to measure the tip-sample force at a given X,Y coordinate, while the user changes one or more physical quantities in a controlled manner. Most often, the quantity that is changed, is either ramped or stepped through a range of values at a given time rate of change that the user controls. The ramped parameter is often the length of the Z-actuator, changing the separation between the sample surface and the probe. The most widely

used AFM spectroscopy mode is Quasi-static AFM Force Spectroscopy (or Contact Mode Force Spectroscopy).

Although STS, Near-field Optical Spectroscopy, and Quasi-static (Contact Mode) AFM Force Spectroscopy are the most widely used SPM spectroscopy modes, others have proven very useful in specialized industrial applications, especially in electrical characterization of semiconductor materials and devices. These involve, for example, applying a bias and measuring a current, for localized I-V curves, or using a resonant capacitance sensor to measure localized C-V curves with the AFM tip.

SPM spectroscopy modes involving Dynamic Mode AFM in which the cantilever is oscillating have been instrumental in understanding the details and the fundamental physics underlying the tip-sample interaction in these modes, namely, Intermittent Contact AFM and FM-AFM.

Electric Force Microscopy and Kelvin Force Microscopy with Agilent MAC Mode III

Electric Force Microscopy (EFM) and Kelvin Force Microscopy (KFM)⁶ are atomic force microscopy (AFM) techniques in which a conductive AFM tip interacts with the sample according to the sample's electrostatic characteristics. EFM is an imaging technique that maps, at each in-plane coordinate pair (X,Y), the out-of-plane (Z) gradient of the electric field emanating from the sample surface. KFM, also an imaging technique, maps the variation of the contact potential between the tip and the sample at each in-plane position. (KFM is also known as surface potential imaging.) Agilent's MAC Mode III control electronics allows truly-simultaneous recording of topography and either EFM or KFM images in a single pass, that is, without the time-consuming process of having to scan twice

(two-pass scanning), once for topography and once for the electrical image. MAC Mode III enables this by incorporating three independently-controlled lock-in amplifiers, one of which is dedicated to the electrical measurement at the same time that another one is used for topography imaging. This arrangement allows the user to choose the frequencies at which the two lock-in amplifiers operate independently from each other, increasing the operational freedom for electrical experiments.⁷ The simultaneous measurement scheme, obviating the need for two-pass scanning, also eliminates the adverse effects on the fidelity of the electrical image that come about due to the drift that the scanner may suffer during the second pass of a two-pass implementation.

⁶ Kelvin Force Microscopy is based on the original work of Sir William Thomson (later, Lord Kelvin), who demonstrated that when two dissimilar conductors are brought into contact, an electric potential develops across the junction. This potential is known as the contact potential difference (CPD), and is determined by work functions of the two materials, which in turn depends on the environment in which the materials are and the contact is made. But in the original Kelvin method and in Kelvin Force Microscopy, the CPD measurement is performed absent perpetual contact between the two conductors. See for example, the article by M. Nonnenmacher *et. al.*, in *Applied Physics Letters*, Volume 58(25), pp. 2921-2923, June 24, 1991.

⁷ For example, the electric signal may be monitored at a frequency twice that of the mechanical signal that drives the AFM cantilever (typically at or near the cantilever's fundamental resonance frequency), and this provides a measure of the gradient of the tip-sample capacitance (C) with z, or dC/dz.

Scanning Microwave Microscope (SMM)

Phase Agilent Technologies' Scanning Microwave Microscope (SMM) Mode is a new Atomic Force Microscope mode that combines the electromagnetic measurement capabilities of a microwave vector network analyzer (VNA) with the nanometer-resolution and Angstrom scale positioning capabilities of an AFM. Agilent VNAs are highly sophisticated characterization instruments that make extremely accurate, calibrated measurements of complex-valued ratios on electromagnetic signals. The ratios are

$$R = \frac{\text{reflected signal}}{\text{incident signal}} \text{ and } T = \frac{\text{transmitted signal}}{\text{incident signal}}$$

The incident signal is generated and controlled inside the VNA; as a result, the ratios R and T are not merely relative, but referenced to the well-known, accurately quantified incident signal. This measurement capability, delivered to the apex of an AFM tip makes the SMM the only AFM in its class, enabling calibrated, traceable measurements of electrical properties such as impedance and capacitance, with the high spatial resolution that is the hallmark of a well designed, well-constructed AFM. A VNA has two operational modes: transmission and reflection. In the reflection (alternatively, transmission) mode, the VNA

measures the magnitude and phase characteristics of the DUT by comparing the signal that reflects off (alternatively, transmits through) the device with the stimulus signal. Each mode enables measurement of several useful parameters. In the reflection mode, the VNA can measure (among other things) the impedance of the DUT. Scanning Impedance Microscopy is a major application of the SMM as is Scanning Capacitance Microscopy.