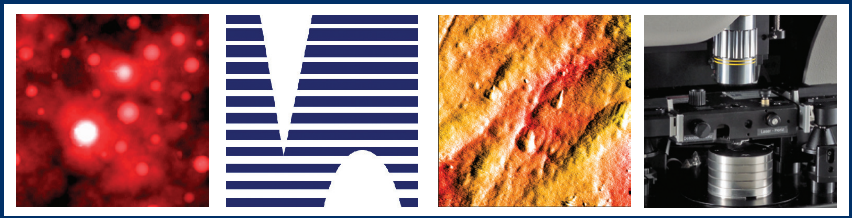

Introduction to Atomic Force Microscopy

Theory Practice Applications

Paul E. West, Ph.D.

CHAPTER 1

Introduction

Typically, when we think of microscopes, we think of optical or electron microscopes. Such microscopes create a magnified image of an object by focusing electromagnetic radiation, such as photons or electrons, on its surface. Optical and electron microscopes can easily generate two-dimensional magnified images of an object's surface, with a magnification as great as 1000X for an optical microscope, and as large as 100,000X for an electron microscope. Although these are powerful tools, the images obtained are typically in the plane horizontal to the surface of the object. Such microscopes do not readily supply the vertical dimensions of an object's surface, the height and depth of the surface features.

Unlike traditional microscopes, the AFM does not rely on electromagnetic radiation, such as photon or electron beams, to create an image. An AFM is a mechanical imaging instrument that measures the three dimensional topography as well as physical properties of a surface with a sharpened probe, (see Figure 1-1).

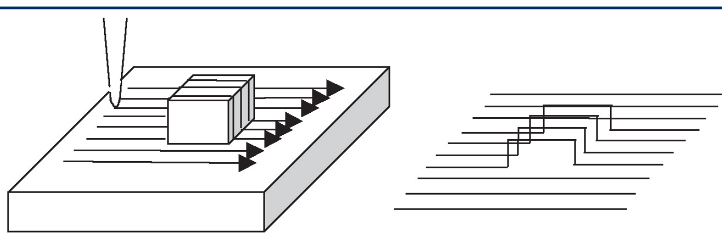

FIGURE 1-1 In the AFM, a sharp probe is scanned across a surface, left, and by monitoring the motion of the probe from each pass across the surface, a 2-D line profile is generated. Then the line profiles are combined to create a three dimensional image of the surface, right.

The sharpened probe is positioned close enough to the surface such that it can interact with the force fields associated with the surface. Then the probe is scanned across the surface such that the forces between the probe remain constant. An image of the surface is then reconstructed by monitoring the precise motion of the probe as it is scanned over the surface. Typically the probe is scanned in a raster-like pattern.

In an AFM the probe is very sharp, typically less than 50 nanometers in diameter and the areas scanned by the probe are less than 100 um. In practice the heights of surface features scanned with an AFM are less than 20 um. Scan times can range from a fraction of a second to many 10's of minutes depending on the size of the scan and the height of the topographic features on a surface. Magnifications of the AFM may be between 100 X and 100,000,000 X in the horizontal (x-y) and vertical axis.

Figure 1-2 illustrates the block diagram of an atomic force microscope. In the microscope, the force between a nanoscopic needle and the surface is measured with a force sensor, the output of the force sensor is then sent to a feedback controller that then drives a Z motion generator. The feedback controller uses the force sensor output to maintain a fixed distance between the probe and the sample. X-Y motion generators then move the probe over the surface in the X and Y axis. The motion of the probe is monitored and used to create an image of the surface.

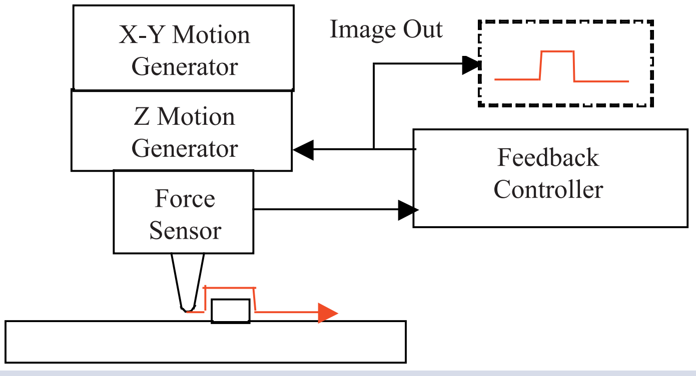

FIGURE 1-2 Basic block diagram of an AFM.

The force sensor in an atomic force microscope is typically constructed from a light lever, see (Figure 1-3). In the light lever, the output from a laser is focused on the backside of a cantilever and reflected into a photo-detector with two sections. The output of each of the photo-detector sections is compared in a differential amplifier. When the probe at the end of the cantilever interacts with the surface, the cantilever bends, and the light path changes causing the amount of light in the two photo-detector sections to change. Thus the electronic output of the light lever force sensor, So, is proportional to the force between the probe and sample.

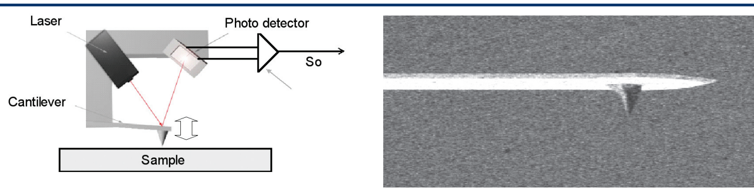

FIGURE 1-3 Illustration of the light lever force sensor (left). An AFM SEM image of the cantilever/probe used in an AFM force sensor (right).

Although the AFM is capable of extreme magnification, it is not a large instrument. An AFM that is capable of resolving features as small as a few nanometers can be easily installed in a laboratory on a desk top. The greatest deterrent to high magnification with the AFM is often environmental vibrations that cause the probe to have unwanted vibrations.

Although the AFM is an amazing instrument for visualizing and measuring nanometer scale features, it has several characteristics that make it unique. They are:

a) Built-in, Atomic-scale Sensitivity – Most measuring instruments become larger when greater sensitivity is required. With an AFM the sensitivity is built-in at the nanometer or atomic scale. Thus to make the instrument more sensitive, there is no need to make it larger.

b) Fabrication Technology – An AFM may be used for rapidly making changes in surface structures at the nanometer scale. Such changes can be made for a fraction of the amount it would cost with traditional technologies such as e-beam or photolithography.

c) Motion Control – Precise motion control technology is required to accurately scan and position the probe in an AFM at the nanometer scale. Such accurate motion control technology allows cost effective motion control at a level not achievable with other methods.

These three unique characteristics may be applied to other technological and scientific areas such as data storage, genetic engineering and nano-robotics.

1.1 History of AFM

Magnification of the vertical surface features of an object, those features leaving the horizontal plane and extending in the vertical direction, have historically been measured by a stylus profiler. An example of an early profiler is shown in Figure 1-4. This profiler, invented by Schmalz[1] in 1929, utilized an optical lever arm to monitor the motion of a sharp probe mounted at the end of a cantilever. A magnified profile of the surface was generated by recording the motion of the stylus on photographic paper. This type of "microscope" generated profile "images" with a magnification of greater than 1000X.

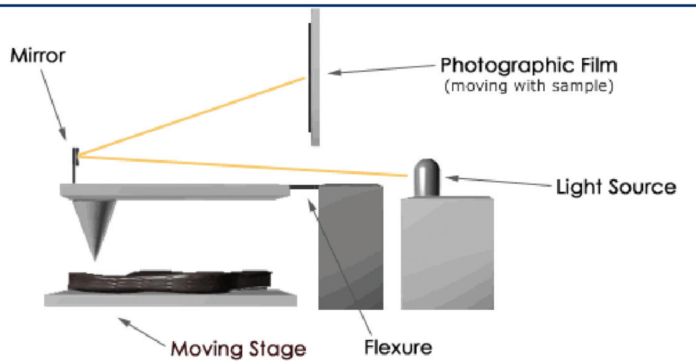

FIGURE 1- 4 Light Lever design used for one of the early designs of a surface profiler in the 1920's. This profiler had a vertical resolution of approximately 25nm.

A common problem with stylus profilers was the possible bending of the probe from collisions with surface features. Such "probe bending" was a result of horizontal forces on the probe caused when the probe encountered large features on the surface. This problem was first addressed by Becker[2] in 1950 and later by Lee[3]. Both Becker and Lee suggested oscillating the probe from a null position above the surface to contact with the surface. Becker remarked that when using this vibrating profile method for measuring images, the detail of the images would depend on the sharpness of the probe.

In 1971 Russell Young[4] demonstrated a non-contact type of stylus profiler. In his profiler, called the topographiner, Young used the fact that the electron field emission current between a sharp metal probe and a surface is very dependent on the probe sample distance for electrically conductive samples. In the topographiner (see Figure 1-5), the probe was mounted directly on a piezoelectric ceramic used to move the probe in a vertical direction above the surface. An electronic feedback circuit

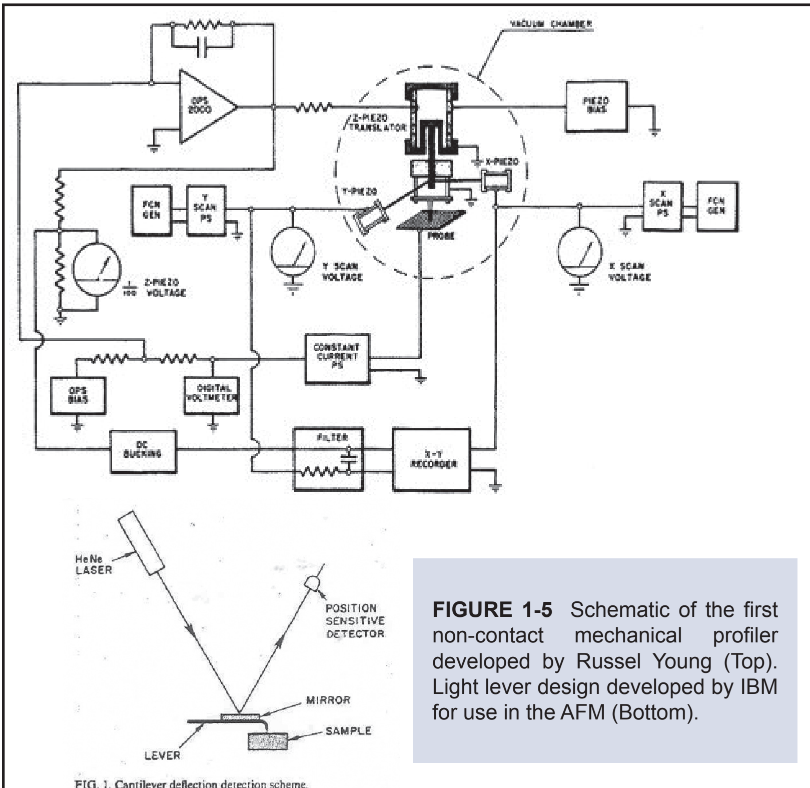

FIGURE 1-5 Schematic of the first non-contact mechanical profiler developed by Russel Young (Top). Light lever design developed by IBM for use in the AFM (Bottom).

monitoring the electron emission was then used to drive the piezoceramic and thus keep the probe sample spacing fixed. Then, with piezoelectric ceramics, the probe was used to scan the surface in the horizontal (X-Y) dimensions. By monitoring the X-Y and Z position of the probe, a 3-D image of the surface was constructed. The resolution of Young's instrument was controlled by the instrument's vibrations.

In 1981 researchers at IBM were able to utilize the methods first demonstrated by Young to create the scanning tunneling microscope (STM). Binnig and Rohrer[5] demonstrated that by controlling the vibrations of an instrument very similar to Young's topographiner, it was possible to monitor the electron tunneling current between a sharp probe and a sample. Since electron tunneling is much more sensitive than field emissions, the probe could be used to scan very close to the surface. The results were astounding; Binnig and Rohrer[6] were able to see individual silicon atoms on a surface. Although the STM was considered a fundamental advancement for scientific research, it had limited applications, because it worked only on electrically conductive samples.

A major advancement in profilers occurred in 1986 when Binnig and Quate[7] demonstrated the Atomic Force Microscope. Using an ultra-small probe tip at the end of a cantilever, the atomic force microscope could achieve extremely high resolutions. Initially, the motion of the cantilever was monitored with an STM tip. However, it was soon realized that a light-lever, see Figure 1-5, similar to the technique first used by Schmalz, could be used for measuring the motion of the cantilever. In their paper, Binnig and Quate proposed that the AFM could be improved by vibrating the cantilever above the surface.

The first practical demonstration of the vibrating cantilever technique in an atomic force microscope was made by Wickramsinghe[8] in 1987 with an optical interferometer to measure the amplitude of a cantilever's vibration.

Using this optical technique, oscillation amplitudes of between .3 nm and 100 nm were achieved. Because the probe comes into close contact with the surface upon each oscillation, Wickramsinghe was able to sense the materials on a surface. The differences between photo-resist and silicon were readily observed.

1.2 Comparison of AFM to Other Microscopes & Instruments

The AFM can be compared to traditional microscopes such as the optical or scanning electron microscopes for measuring dimensions in the horizontal axis. However, it can also be compared to profilers for making measurements in the vertical axis to a surface. One of the great advantages of the AFM is the ability to magnify in the X, Y and Z axes.

Figure 1-6 shows a comparison between several types of microscopes and profilometers. One of the limiting characteristics of the AFM is that it is not practical to make measurements on areas greater than 100 um. This is because the AFM requires mechanically scanning the probe over a surface:

(1000X for 100um to 10 cm) (1,000,000X 100nm to 10 cm)

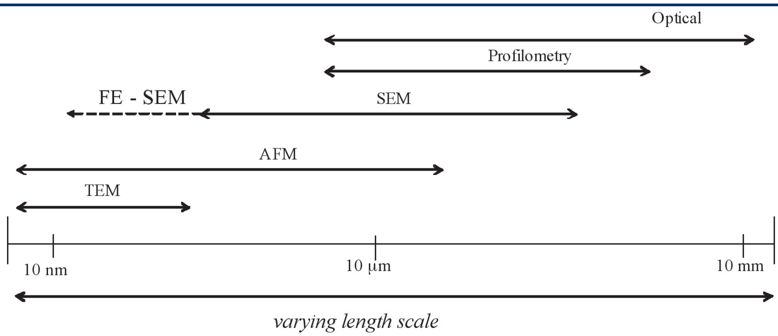

FIGURE 1-6 Comparison of the imaging “length scale” of many types 2-D and 3-D profiling and imaging instruments. An AFM is capable of resolving features in the dimensions of a few nanometers with scan ranges up to a hundred microns.

When compared to a profiler, the AFM has a greater X-Y resolution because in the AFM the probe is sharper. Profilers can have high vertical resolutions, as low as .05 nm. However, the bandwidth of the profiler measurements is much lower than an AFM. To achieve a resolution of .05 nm a profile has a bandwidth of approximately .1 Hz. The AFM bandwidth for the equivalent measurement is between 5 kHz and 10 Khz.

The length scale of an optical microscope overlaps nicely with an AFM. Thus, an AFM is typically combined with an optical microscope and with this combination it is possible to have a field of view dynamic range from mm to nm. In practice, an optical microscope is typically used for selecting the location for AFM scanning.

The AFM is most often compared with the electron beam techniques such as the SEM or TEM. In general, it is easier to learn to use an AFM than an SEM because there is minimal sample preparation required with an AFM. With an AFM, if the probe is good, a good image is measured. A comparison of the some of the major factors follows:

	SEM / TEM	AFM
Samples	Must be conductive	Insulating / Conductive
Magnification	2 Dimensional	3 Dimensional
Environment	Vacuum	Vacuum / Air / Liquid
Time for image	0.1 - 1 minute	1 - 5 minute
Horizontal Resolution	0.2 nm (TEM) 5 nm (FE-SEM)	0.2 nm
Vertical Resolution	n/a	.05 nm
Field of View	100 nm (TEM) 1 mm (SEM)	100 um
Depth of Field	Good	Poor
Contrast on Flat Samples	Poor	Good

FIGURE 1-7 Comparison of an AFM and SEM.

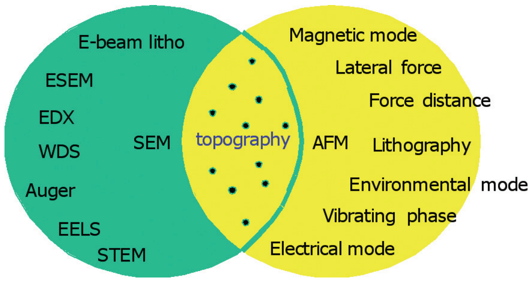

FIGURE 1-8 Both the AFM and SEM measure topography. However, both types of microscopes can measure other surface physical properties. The SEM is good for measuring chemical composition and the AFM is good for measuring mechanical properties of surfaces.

SEM/TEM instruments are capable of doing much more than topography measurements. For example, electron beam instrumentation can do EDX measurements or even electron beam initiated lithography. Likewise, the AFM can make many types of measurements other than AFM topographical measurements. For example, AFM instruments can make thermal, magnetic and electric field maps of a surface. Like the SEM/TEM, an AFM can also initiate lithographic changes on a surface.

Although the time required for making a measurement with the SEM image is typically less than an AFM, the amount of time required to get meaningful images is similar. This is because the SEM/TEM often requires substantial time to prepare a sample. With the AFM, little or no sample preparation is required. Figure 1-9 shows the comparison between a TEM image of nano-particles and the AFM image of the same nanoparticles.

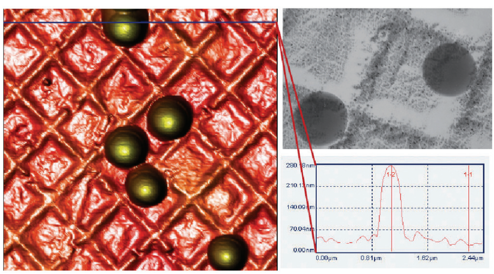

FIGURE 1-9
AFM image, color, and TEM, grayscale, of 500 nm diameter particles. A line profile from the AFM image shows the height of the particles.

In comparison with an optical microscope and the SEM/TEM an AFM is more difficult to use than the optical microscope and easier to use than the SEM/TEM. Also, the AFM is typically more expensive than the optical microscope and less costly than an SEM/TEM. Figure 1-10 compares the relative time and cost for optical, AFM, and SEM/TEM microscopes.

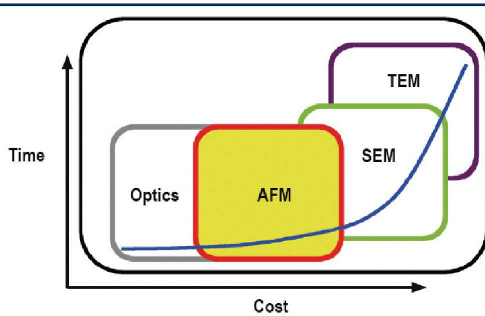

FIGURE 1-10
Comparison of the time for measurements and instrumentation cost of optical, AFM, and SEM/TEM microscopes.

Lastly, an optical microscope requires the least amount of laboratory space, while the SEM/TEM requires the most amount of laboratory space. An AFM is in the middle of these two.

Finally, in comparison to an optical profiler, the AFM is more difficult to use. This is because the optical profiler does not need any adjustments. However, the AFM requires adjustments of the scan speed and the feedback control parameters.

1.3 Enabling Nanotechnology

Approximately 20 years ago scientists and engineers began discussing a technological revolution that would be as dramatic and far-reaching to society as the industrial revolution - the nanotechnology revolution. At first the primary promoters of the nanotechnology revolution were considered eccentric at best, and a little crazy at worst. However, their ideas and visions are becoming accepted by the mainstream intellectual, scientific and engineering communities. Recently, governments and major corporations around the world have committed several billion dollars per year for the advancement of nanotechnology and nanoscience research and development.

Atoms and Molecules

The systematic study, manipulation, and modification of atoms and molecules having nanometer-sized dimensions began several hundred years ago. Society has benefited greatly because chemists can use chemical reactions to combine several types of atoms to create new types of molecules. With the advent of quantum physics, physicists, chemists and biologists routinely studied the spectra of atoms and molecules. Biochemists discovered the usefulness of all types of molecules from proteins to enzymes to DNA several decades ago.

Until recently however, working with and controlling atoms and molecules was limited to large quantities of these nanometer-sized objects. Realistically, chemists would modify hundreds of trillions of molecules in a typical chemical reaction. When chemists synthesize new molecules, they make them in large quantities by using macroscopic methods such as heat to initiate chemical reactions. Biologists can identify and create new types of genetic material, but only on a large number of molecules.

So what's new?

The nanotechnology revolution is being driven by a number of developments, ideas, and technical advancements. The primary driving forces behind the nanotechnology revolution are instruments that measure and manipulate atoms and molecules.

The invention of the Scanning Tunneling Microscope permitted us for the first time to see single atoms on a surface. Before this, using techniques based on electromagnetic radiation, it was possible to view and create images of lattices of many molecules. For example, with x-ray techniques it is possible to recreate the positions of atoms in a complex matrix or lattice. With tunneling electronic microscopes (TEM) it is possible to directly image atoms in a lattice. However, these techniques rely on the scattering of electromagnetic radiation from a collection of atoms, and thus cannot see single atoms.

Another important innovation is the laser "tweezer". By using the momentum of photons it is possible to isolate in a single location collections of several hundred molecules or atoms. The possibility of isolating a few molecules, or even a few hundred molecules, was not considered possible before this invention .

The drive to make smaller computer chips & higher density information storage

Moore's law, popularized in the late 20th century, dictates that there is a relationship between the size of electronic devices such as transistors and time. This relationship has been very effective in predicting advances in the world of microelectronics for almost thirty years. However, physicists are predicting that Moore's law will begin breaking down when the size of electronic devices becomes less than 100 nanometers. There is a great effort to discover new methodologies for creating electronic devices with dimensions that are less than 100 nanometers.

The storage of information is considered an essential advancement of modern civilization. At first, recording information and ideas on written paper was a great achievement; books and newspapers allowed the flow of knowledge and information throughout the world. Today information is stored digitally and transmitted electronically. Digital bits with dimensions of less than a micron are stored on magnetic disks and compact discs. There is an ever-increasing need to store information in smaller spaces and transmit information with faster methodologies.

Emerging belief that it is possible to mimic the mechanisms of biology

Researchers in the fields of life sciences discovered over the past few decades that there are many fundamental mechanisms that facilitate the recreation and support of all life forms. At a distance these mechanisms can be characterized as machines or engines. They absorb energy and in a very efficient way cause events to occur. For example, a virus will permeate a cell and then integrate with the genetic material of the cell.

Presently, we can observe these activities on a macroscopic scale. In many cases we do not understand how they work or why they work. But there is a belief that we can understand, emulate, and even use these fundamental activities or machines that occur in biological systems.

Creation of mechanical devices having nanometer tolerances and motions (MEMS)

To a great extent, the industrial revolution occurred because it became possible to shape mechanical objects, and thus create new types of machines. Before the industrial revolution, it was possible to routinely make objects that had dimension on the order of a few hundredths of an inch. An artist could paint pictures; a potter could make dishes and pots. After the industrial revolution, it was possible to routinely make machines that had tolerances of a few thousands of an inch (25 to 100 microns). Of course this led the way to the invention of the steam engine, railroads, the car, and airplane transportation.

With MEMS technology it is now possible to use machining technologies to create machines that are less than the size of a human hair. This ability is presently used in the sensors that activate air bags in cars, set the frequency of computers, and allow digital projection.

Nanoscience

Applying the scientific method to further understand the behavior of atoms and molecules at the nanometer scale will push forward the frontiers of human knowledge. Currently our vision of the nano-world is based only on evidence that we collect from the macroscopic world that we live in. Presently biologists, chemists, physicists and engineers only have a mental picture of what is occurring at the nanometer scale. In fact, only very recently have they actually seen or directly observed nano-events.

As an analogy, suppose you were presented with a gift that was in a box and wrapped with paper. In an effort to guess what is in the package, you could shake it or maybe drop it. Based on how the package behaves under this "interrogation", you may get an idea of what is in it (i.e. is it heavy, does it make a noise?). With the Nano-R™ evolution, scientists will be able to open the package... and really see what is inside. With new ideas and methods, scientists are beginning to further understand how a single atom or molecule behaves. Even more interesting is the direct understanding of how collections of two or three or even a dozen atoms or molecules behave.

Nanotechnology

The fundamental knowledge gained through nanoscience and developments in nanotechnology will certainly accelerate over the next several decades. With the control of materials at the nanometer dimension, engineers are already able to create new types of products and services. The smallest transistors we make today in a factory are about 130 nanometers wide. With future nanotechnology advancements engineers will be able to make chips that have transistors that are two or three nanometers wide. Today, cosmetic manufacturers use liposomes with diameters of a few tens of nanometer to reduce the dehydration of skin. We expect the nanotechnology revolution will result in the creation of new types of products and services that will greatly benefit our lives.

What is possible?

When the ideas and concepts that are discussed as part of the nanotechnology revolution are fully implemented, what is possible? Many of the possible advancements that are discussed today seem like science fiction.

We can only imagine what is possible. Imagine . . .

1. All of recorded history will fit in a package that will fit in our pockets. This includes all written documents, all music and all movies.
2. Our world will be safer because computers and sensing systems that fit in a package the size of a pill will be able to warn us of dangers.
3. Life will be extended because we can create systems and modules that replicate the functions and systems in our bodies.
4. New types of "quantum computers" will make calculations billions of times faster than today's digital computers.
5. We can create new types of molecules with the mechanical assembly of chemical systems instead of today's assembly by thermodynamic chemical reactions.

What is the Atomic Force Microscopes Contribution to Nanotechnology?

Measurement

An atomic force microscope (AFM) creates a highly magnified three dimensional image of a surface. The magnified image is generated by monitoring the motion of an atomically sharp probe as it is scanned across a surface. With the AFM it is possible to directly view features on a surface having a few nanometer-sized dimensions including single atoms and molecules on a surface. This gives scientists and engineers an ability to directly visualize nanometer-sized objects and to measure the dimensions of the surface features.

With an atomic force microscope it is possible to measure more than the physical dimensions of a surface. This is because there is a "physical" interaction of the probe with a surface. By lightly pushing against a surface with the probe, it is possible to measure how hard the surface is. Also the ease by which the probe glides across a surface is a measure of the surface "friction".

Modification

Just as a pen is used for writing on a paper's surface, it is possible to write on a surface with an atomic force microscope. This new type of "lithography" results in a completely new method for making surface modifications at the nanometer scale. It is already possible to modify surfaces by physically scratching the surface, by directly depositing molecules on a surface, and by using electric fields to modify surfaces. Presently this use of the AFM is in a very exploratory phase, but showing tremendous promise. One of the important technological issues that must be solved is the writing speed of the AFM lithography systems.

Manipulation

With an AFM probe it is possible to directly move objects across a surface. The objects may be pushed, rolled around, or even picked up by the probe. With such methods it is possible to create nanometer sized objects. One of the important aspects of using an AFM for direct manipulation is the user interface that is used for generating the motions of the probe. There are interfaces that measure the locations of particles, such as microspheres on a surface, and then automatically move the spheres into a pre-established location. In another type of interface, called the nanomanipulator, the motion of the probe follows the motion of your hand. When you move your hand up and down, the probe moves up and down. Such an interface also allows users to "feel" and "touch" a surface.

The Nanotechnology Timeline

Nanotechnology has had impact on our lives for 20 to 30 years. As early as 1970 there were many products that relied on nanometer sized components to operate, such as in the semiconductor industry. However, the time line for radically new, social structure changing advancements is still many years away. Although it impossible to predict the future, Figure 1-11 illustrates the time frames for the development of new and advanced products from nanotechnology innovations.

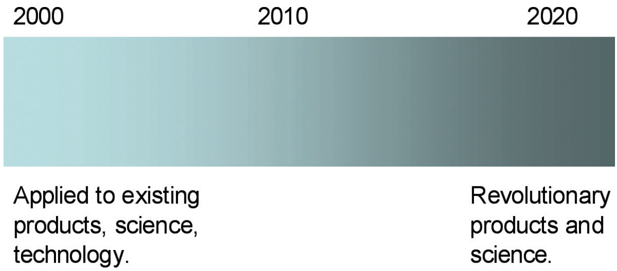

FIGURE 1-11 Timeline for the integration of nanotechnology and nanoscience into products. In the short term efforts are underway to enhance existing products with nanotechnology concepts. Far in the future new types of products not presently imagined will be created from nanotechnology

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CHAPTER 2

AFM Instrumentation

An AFM is a deceptively simple instrument that requires a considerable amount of engineering construct such that the AFM is capable of measuring images with nanometer resolution. The basic components of an AFM are a computer, control electronics, and a stage.

FIGURE 2-1 Photograph of a table top AFM illustrating the major components. They are a microscope stage, computer, electronic controller, computer monitor, and optical microscope monitor. The trackball is used for moving the sample stage in the X-Y axis. Most of the AFMs in use in laboratories around the world are table top units such as this. Resolution can often be enhanced by placing the microscope stage on a vibration isolation table.

The functions of each of the microscope's components is:

Computer: Software in the computer is used for acquiring and displaying AFM images. Also, software for processing and analyzing AFM images typically resides in the computer.

Control Electronics: The control electronics generate the electronic signals required for moving stage components such as the Z motors and the XYZ scanner. The control electronics also digitize the images measured in the stage so that they can be displayed by the computer.

Stage: An AFM stage is where the sample is placed when an image is measured. Typically, the AFM stage includes an AFM scanner and an optical microscope, along with motion control systems for moving the scanner relative to a sample in the X-Y-Z axis.

An AFM may be constructed for laboratory use and easily fit on a table top; it may also be constructed to accommodate large samples such as semiconductor wafers. An AFM capable of holding wafers can be large and have a volume of several cubic meters. Regardless of the size of the AFM, they all have the similar components. Often the electronics are separated from the stage because fans used to cool the electronics cause unwanted vibrations in the stage which reduce the resolution of the AFM.

2.1 Basic Concepts

There are three basic concepts that you must be familiar with to understand the operation of an AFM. These are piezoelectric transducers, force transducers, and feedback control.

Piezoelectric Transducers

Piezoelectric materials are electromechanical transducers that convert electrical potential into mechanical motion. Piezoelectric materials are naturally occurring and may be crystalline, amorphous or even polymeric. When a potential is applied across two opposite sides of the piezoelectric, it changes geometry. The magnitude of the dimensional changes depends on the material, the geometry of the device, and the magnitude of the applied voltage.

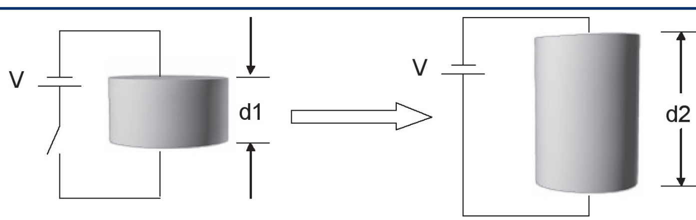


FIGURE 2-2 A piezoelectric disc will expand radially when a voltage potential is applied to the top and bottom electrodes. The disc will change shape such that volume is preserved.

Piezoelectric materials are available in a number of sizes and shapes. Typically, the expansion coefficient for a single piezo shape is on the order of 0.1 nm per applied volt. Thus, if the voltage used to excite the piezo material is 2 Volts, then the material will expand approximately 0.2 nm, or the diameter of a single hydrogen atom. Piezoelectric materials are used for controlling the motion of the probe as it is scanned across a surface in an AFM. Section 2.2.1 describes in greater detail how piezoelectric materials may be configured to scan a probe in three dimensions.

Force Transducers

The force between a probe and a surface is measured with a force transducer. As illustrated in Figure 2-3, when the probe comes into contact with the surface, the voltage output from the transducer increases. It is important that the output of the transducer be monotonic and increase as a greater force is applied between the probe and surface. Force transducers may be constructed that measure forces as low as 10 picoNewtons between a probe and a surface. There are several types of force sensors that may be used in an AFM (presented in Section 2.2.2).

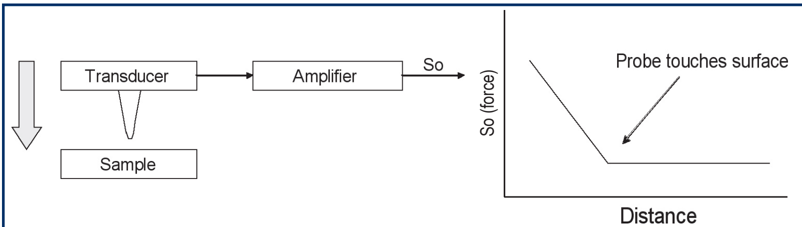


FIGURE 2-3 The force transducer outputs an electronic signal when the probe interacts with forces from the surface. Left: An amplifier outputs a voltage, So. Right: As the probe interacts with the surface, So increases monotonically.

Feedback Control

Feedback control is used in AFM for maintaining a fixed relationship, or force, between the probe and the surface. The feedback control operates by measuring the force between the surface and probe, then controlling a piezoelectric ceramic that establishes the relative position of the probe and surface. Feedback control is used in many applications, Figure 2-4 illustrates the use of feedback control in an oven. Section 2.3 has a more detailed discussion of feedback control methodologies in an AFM.

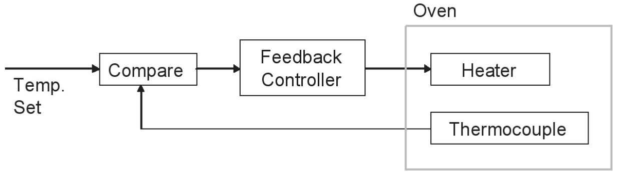


FIGURE 2-4 Feedback control is routinely used to control the temperature in an oven. The heater raises the temperature in the oven until the thermocouple and set point temperature are equal.

AFM Block Diagram

An AFM is constructed using piezoelectric materials, a force transducer and feedback control as illustrated in Figure 2-5. The force transducer measures a force between the probe and surface; the feedback controller keeps the force constant by controlling the expansion of the Z piezoelectric transducer. Then, the X-Y piezo electric ceramics are used to scan the probe across the surface in a raster-like pattern. By monitoring the voltage on the Z ceramic, an image of the surface is measured.

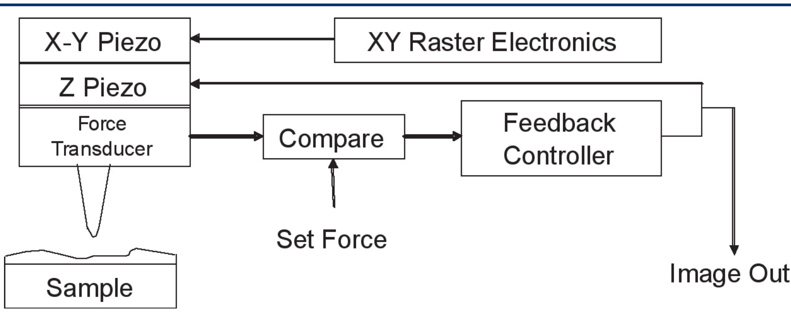


FIGURE 2-5 Block diagram showing the components in an AFM. The image is created by monitoring the voltage that drives the Z piezoelectric ceramic.

There are several challenges that must be met to properly construct an atomic force microscope. They are:

a) A very sharp probe must be constructed so that high resolution images are measured.

b) To get the probe within the scanning range of the surface, a macroscopic translation mechanism must be constructed.

c) The force transducer must have a force resolution of 1 n/N or less so that the probe is not broken while scanning.

d) A feedback controller that permits rapid control so that the probe can follow the topography on the surface must be created.

e) An X-Y-Z piezoelectric scanner that has linear and calibrated motion must be used.

f) A structure must be constructed that is very rigid so that the probe does not vibrate relative to the surface.

g) A high speed computer that can display the images in real time as they are collected must be used.

h) A stage that allows rapid exchange of the probe used for scanning must be created.

2.2 The AFM Stage

Figure 2-6 illustrates the primary components of an AFM stage. There is an AFM scanner that measures the force between the probe and surface and scans the probe over the surface. There is a motion control mechanism, the Z motor, which can move the AFM scanner towards the

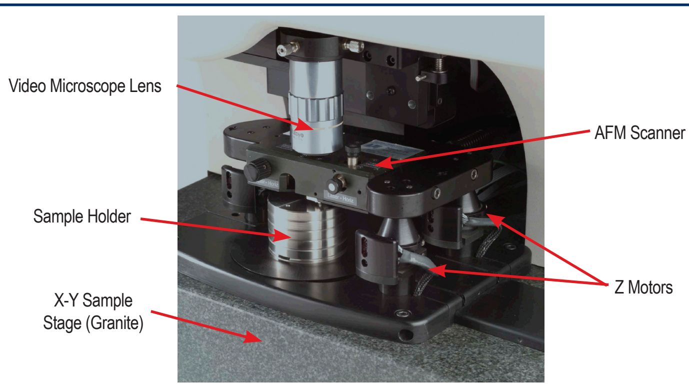


FIGURE 2-6 Photograph of an AFM stage.

sample. There is also an X-Y positioning stage which is not required but is useful for positioning the feature for imaging under the probe as well as an optical microscope for viewing the probe and surface. A mechanical structure is required to support the AFM scanner.

In the construction of the stage it is important that the mechanical loop, that is, all the mechanical components between the probe and surface, be very rigid. If the mechanical loop is not rigid, the probe will tend to vibrate relative to the sample and introduce unwanted noise into the images. In general, if the microscope stage is smaller, it will be less susceptible to external vibrations. Creating a rigid mechanical loop becomes more difficult the larger the sample size is. The highest resolution AFMs tend to be very small so that the mechanical loop is rigid, and the microscope stage is not susceptible to external environmental vibrations (or noise).

In an AFM stage the motion control mechanisms capable of moving several millimeters or greater are designated X,Y, and Z. The motion control mechanisms that are used for moving small distances are designated x,y, and z. This nomenclature is used throughout this document.

It is possible to construct an AFM with two different configurations, as illustrated in Figure 2-7. In the first configuration the sample is scanned and the force sensor is held in one place. In the second configuration, the sample is held fixed and the probe is scanned. The advantage of the probe scanning microscope is that it can be used on any size of sample. However, the construction of a probe scanning microscope is much more difficult.

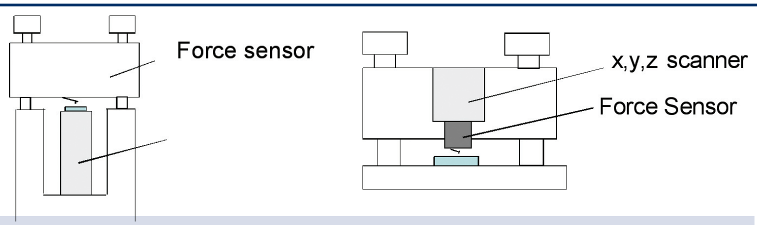


FIGURE 2-7 Left: In a “sample scanning” AFM the sample is mounted on an xyz scanner and the force sensor remains fixed. Right: In the “tip scanning” AFM the sample remains fixed and the probe is scanned. The advantage of a “tip scanning” AFM is that it can scan many sample sizes.

2.2.1 XYZ Scanners

Typically, the scanners used for moving the probe relative to the sample in an AFM are constructed from piezoelectric materials. This is because piezo materials are readily available, easily fabricated in desirable shapes, and cost effective. Scanners in an AFM may be constructed from other types of electromechanical devices such as voice coils. All that is important is that the electromechanical device have accurate positioning.

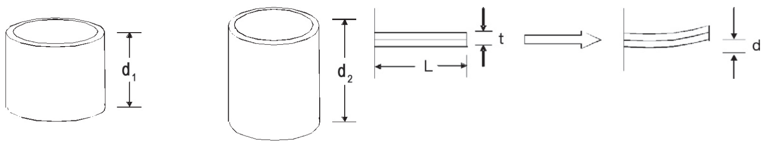
Piezoelectric Materials

The most common types of piezoelectric materials in use for AFM scanners are constructed from amorphous PdBaTiO3. The ceramics may be "hard" or "soft", depending on the formulation. Hard ceramics have smaller coefficients of expansion, but are more linear. Soft ceramic formulations have more non-linearities and have greater expansion coefficients. After fabrication, piezoelectric ceramics are polarized. Polarization may be lost by elevating the piezos to a temperature above their critical temperature or by giving them an over voltage.

Electronically, piezos act as capacitors and store charges on their surface. Capacitances of ceramics may be as large as 100 μfarads. Once a charge is placed on the piezoceramic, the piezoceramic will stay charged until it is dissipated. Electronic circuits used for driving the piezoceramics in an AFM must be designed to drive large capacitive loads.

All piezoceramics have a natural resonance frequency that depends on the size and shape of the ceramic. Below the resonance frequency, the ceramic will follow an oscillating frequency, at resonance there is a 90 degree phase change, and above resonance there is a 180 degree phase change. To a great extent, the resonance frequencies of the piezoelectric ceramics limit the scan rates of atomic force microscopes.

Piezoelectric materials can be fabricated in several shapes such that they have more or less motion. As an example, a disk, as illustrated in Figure 2-8, gets longer and narrower when a voltage is applied. The ceramic changes geometry such that the volume is preserved during extension. Another configuration for a piezoelectric ceramic is a tube, with electrodes on the inside and outside. This configuration gives a lot of motion, and is very rigid. Another configuration is the bimorph, constructed from two thin slabs of piezo material that are polarized in opposite directions. When a voltage is applied the ceramic expands in a parabolic fashion.



Equation 2-1: $d_2 - d_1 \ \alpha \ \frac{d_1}{t} \times V$

Where: V = Voltage applied to PZT

t = Tube thickness

Equation 2-2: $d \ \alpha \ \frac{L^2}{t} \times V$

Where: V = Voltage applied to PZT

t = Tube thickness

FIGURE 2-8 Left: A piezoelectric cylinder has electrodes on the inside and outside so that the potential goes through the wall of the ceramic. When activated, the cylinder elongates. Right: A piezoelectric bimorph has two sheets of piezoelectric material bonded together. The end of the bimorph expands in a parabolic motion when activated.

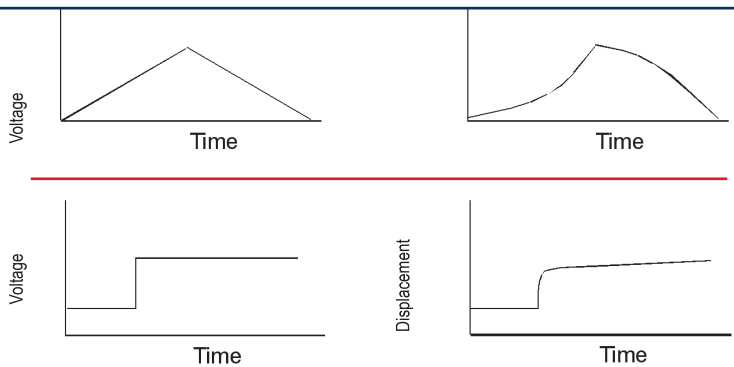


FIGURE 2-9 Top: PZT materials have hysteresis. When a voltage ramp is placed on the ceramic, the motion is nonlinear. Bottom: Creep occurs when a voltage pulse on a PZT causes initial motion followed by drift.

Ideally, the piezoelectric ceramics would expand and contract in direct proportion to the driving voltage. However, piezoelectric materials have two primary non-ideal behaviors, hysteresis and creep. Hysteresis, derived from the word history, causes the ceramic to maintain the shape that it was in. As the ceramic is expanding, there is a negative shaped non-linearity, and as the material is contracting, there is a positive shaped non-linearity. Creep occurs when the ceramic is subjected to a sudden impulse such as a voltage step function. These non-ideal behaviors must be corrected or they cause distortions in AFM images.

Correcting the non-ideal behaviors of piezoelectric ceramics is essential for making accurate measurements with an AFM. Also, hysteresis and creep make it difficult to rapidly make AFM measurements. The non-ideal motions of piezoelectric ceramics may be corrected using open loop or closed loop methods. The corrections are different for the x,y and the z axis.

X,Y axis correction

Open loop techniques require calibration of the AFM scanner to measure the non-linearities. Then the image is corrected using the measured non-linearities. In real time, the voltage used to excite the ceramic may be altered to compensate for the non-ideal behavior. After an image is measured it may also be "corrected" by applying a correction function that was previously created. Open loop techniques are adequate for correcting non-linearity when making measurement with pre-determined scan ranges and speeds. However, open loop techniques cannot correct for problems associated with creep.

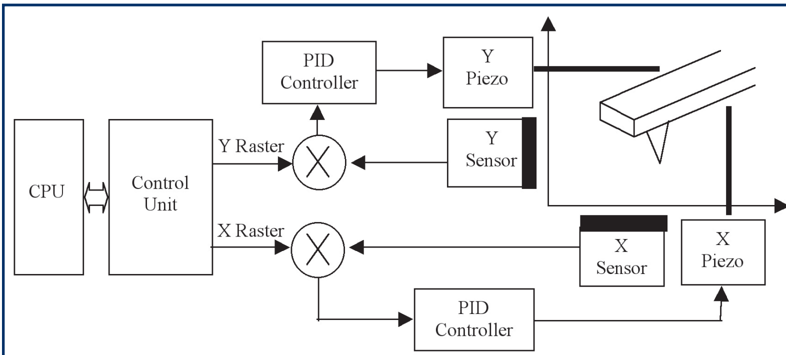


FIGURE 2-10 Block diagram of an x-y closed loop scanner configuration. As the piezo expands, the motion sensor measures the motion. Output from the motion sensors is used to correct the motion of the piezo.

Another technique for correcting hysteresis and creep is to use an external sensor to correct for both. An external position sensor can be used in an open loop or closed loop design. In the open loop configurations, the position of the scanner is measured; then the image is corrected after it is measured. In the closed loop configuration, the motion of the probe is corrected in real time with a feedback electronic circuit. Figure 2-10 shows the use of external sensors in a closed loop design.

Many types of position sensors may be used for correcting the unwanted characteristics in piezoelectric materials. The position sensor must be small in size, stable over long time periods, easily calibrated, have very low noise floors, and be easily integrated into a scanner. Several types of position sensors are available including light based sensors, induction sensors, and capacitance sensors.

Optical Sensors

One of the first position sensors employed in a probe microscope scanner used a light source, a moving knife edge, and a photo-detector, illustrated in Figure 2-11. As the knife edge moves across the light source it reduces the amount of light reaching the photo-detector, which gives a measure of the scanner's motion. Other types of light-based motion sensors include using a pinhole above a position sensitive detector and a light lever. Each of these light-based designs requires a high gain amplifier.

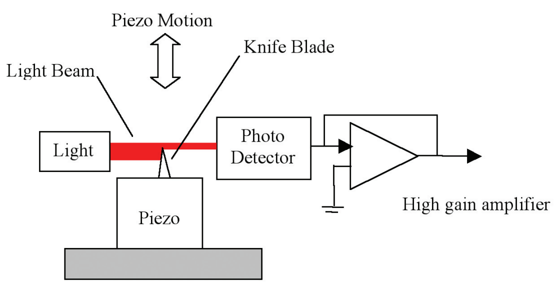


FIGURE 2-11 Design of motion sensor using a light source and photodetector. As the knife edge is moved up and down by the piezo, the amount of light reaching the photodetector changes.

The primary advantage of the light-based position sensors is that the parts required for construction are relatively inexpensive to purchase. There are, however, many disadvantages. Disadvantages include:

a) The sensor is not inherently calibrated

b) Misalignments of the light source and scanner can cause great errors.

c) Linearity can depend on the "integrity" of the light beam.

d) Repair is costly.

e) Integration into an SPM scanner in 3 axes is difficult.

f) Measurement is made at near DC so there is a large 1/f noise.

g) Light sources can cause thermal drift in the scanner.

h) High gain amplifiers with noise and drift are required.

Items a-e make it difficult and cumbersome to use light based techniques in SPM scanners. Items f and g affect the performance of the scanner. The noise in the sensing system requires the operator to turn off the scanner when zooming in to small scan areas. Then, the zoomed image shows unwanted image artifacts derived from hysteresis and creep. Further, when transitioning from closed loop to open loop, the position of the zoomed image can change. Typically scanners with light based systems show thermal drift in the images caused by the light source.

Inductive Sensors

Inductive motion sensors are far better for measuring the displacement of the piezoelectric ceramics in SPM scanners. They have low noise, are inherently calibrated, and are very robust. Inductive scanners are constructed from a coil (see Figure 2-12).

In an inductive sensor, an AC current flowing in a coil causes the field of one winding to add to the field of the next winding. The fields pulsate in turn generating a pulsating electromagnetic field surrounding the coil. Placing the coil a nominal distance from an electrically conductive "target" induces a current to flow on the surface and within the target (because of the circular pattern, the induced current is called an "eddy current"). The induced current produces a secondary magnetic field that opposes and reduces the intensity of the original field – or a "coupling effect." The strength of the electromagnetic coupling between the sensor and target depends upon the gap between them.

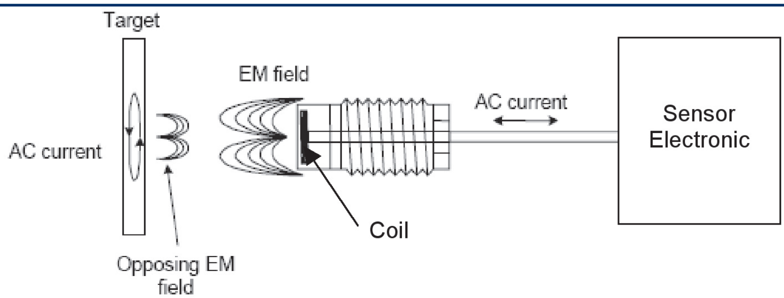


FIGURE 2-12 Inductive motion sensors measure the motion of a target as it moves relative to the sensor. Magnetic fields emanate from the front of the sensor.

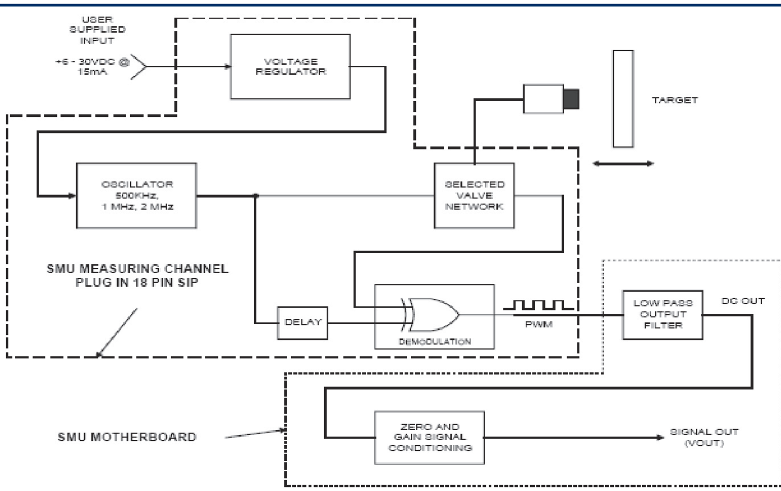


FIGURE 2-13 The sensor system is comprised of a sensor, signal conditioning electronics with an integral DC input, and a system voltage output cable. Unlike the light based motion sensors, no high gain amplifier is required.

The inductive motion sensors used by Pacific Nanotechnology are based on eddy current inductive technology. The sensors rely upon impedance variations in the sensor coil, and detection and demodulation in a phase detection circuit based on pulse width modulation techniques (PWM). The sensors have electronics that have a very low system noise. The sensors do not require a high gain amplifier because PWM is used. The noise that is present is almost “white” in character and diminishes as the bandwidth narrows towards DC. Inductive sensor systems can be optimized for temperature stability and linearity.

In comparison to the light based position sensors, the inductive sensors are ideal for use in SPM scanners. Inductive scanners:

- are modular and easily replaced
- are small and can be easily integrated in three scanning axes
- use a modulated signal and have low 1/f noise
- are stable and require calibration only once at the factory
- have nearly linear output

Other Sensors

Several other types of sensors may be used for correcting the scan errors associated with PZT materials. Capacitance-based motion sensors are common primarily because the electronics for capacitance sensors are very sensitive. Additionally, temperature-based strain gauges may be used. Strain gauges can be attached directly to the piezoelectric material or they may be attached to a structure which flexes when the piezoceramic expands.

Calibration

One of the advantages of closed loop scan correction is that the scanner can be calibrated. Such calibrations can give very precise and accurate motion control. However, the calibration procedure can be very time consuming. Some of the motion sensors, such as the optical based sensor, are non-linear and require constant re-calibration. Other types, such as the inductive sensors are reasonably linear and rarely require calibration.

Zoom to Feature

One of the problems with AFM scanners with open loop or no scan correction is that they can be very difficult to zoom from a large scan range to a smaller scan range (called zoom-to-feature). Without scan linearization, zooming from a large scan range to a smaller range can take several scans. However, with the scan calibration sensors, zooming to a specific scan location requires no intermediate scans.

z Axis Correction

Correction of hysteresis and creep in the z axis is different than the correction in the xy axis. This is because the xy axis motions are predetermined and the z axis motion is nondeterministic , and depends on the surface topography of the sample being scanned. It is not possible to predict the surface topography, so closed loop methods will not work.

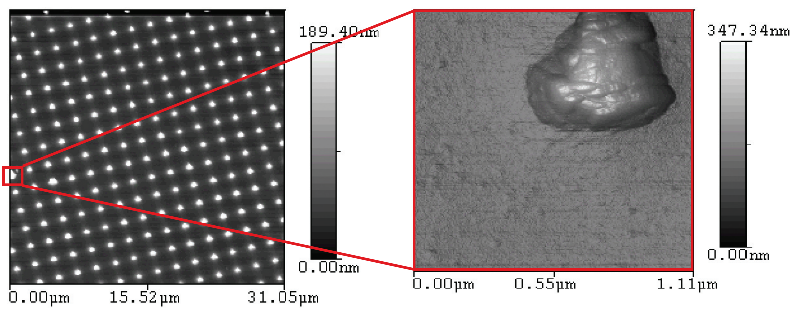


FIGURE 2-14 With a scan calibration sensor, it is possible to scan from a large scan area to a small scan area rapidly. The effects of hysteresis and drift are minimized.

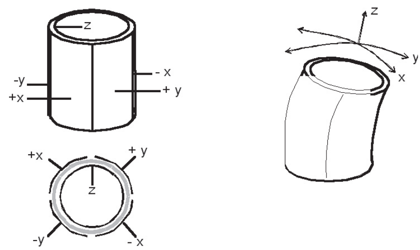
Three dimensional xyz scanners

Piezoelectric ceramics must be configured so that they can move the probe, or sample, in the X,Y and Z axis. There are a few standard configurations that are used in AFM instruments. They are the tripod, the tube, and flexures (see Figure 2-15). Each of these designs may be configured for more or less motion, depending on the application for which the scanner is being used. It is also possible to create scanners that use a combination of any of the 3 basic designs.

Equation 2-3: $\Delta x = \Delta y \; \alpha \; \frac{L^2}{t} \times V$

$\Delta z \; \alpha \; \frac{L}{t} \times V$

Where: V = Voltage applied to PZT
$\Delta x, \Delta y, \Delta z$ = Motions in x, y, z directions



Equation 2-4: $\Delta x \; \alpha \; \frac{L_2}{L_1} \times V$

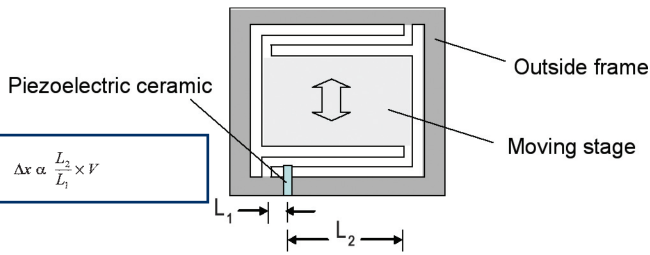


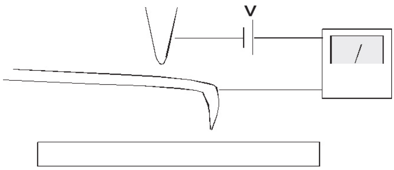
FIGURE 2-15 Top: A PZT tube is configured such that it moves in the x-y-z axis. Four electrodes on the outside are used for the x-y axis motion, and the inner electrode is used for the z axis motion. Bottom: A flexure scanner operates by pushing on a flexure with a PZT. In this design, the ceramic pushes against the flexure which then causes the stage to move. There is a gain in the motion given by the ratio of L_2/L_1.

2.2.2 Force Sensors

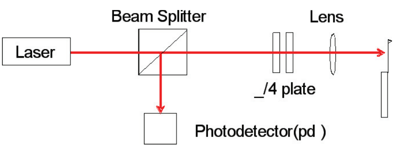
The force sensor in an AFM must be able to measure very low forces. This is because, if a small probe is used, the pressure, force/area, must be small so that the probe is not broken. A number of different force sensors have been tested and demonstrated to work with an AFM. Several of the force sensor designs are illustrated in Figure 2-16.

The light lever, used routinely for measuring minute motions in scientific instrumentation, was first demonstrated in 1988. With the advent of microfabricated cantilevers the Light Lever AFM (LL-AFM) became the most widely used design for the force sensor in an AFM.

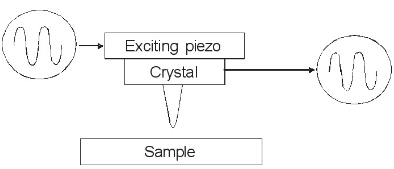
The most widely used force sensor for AFM's is the light lever sensor. However, crystal sensors are rapidly gaining acceptance. The following sections cover the design and implementation of light lever and crystal force sensors.



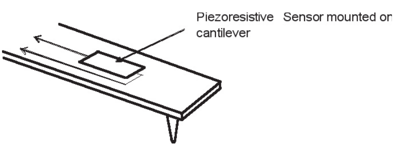
Scanning tunneling microscope: In the original AFM built in 1985 a scanning tunneling microscope tip was use to measure the motion of a cantilever. Although this technique was viable, implementation and operation were very difficult.



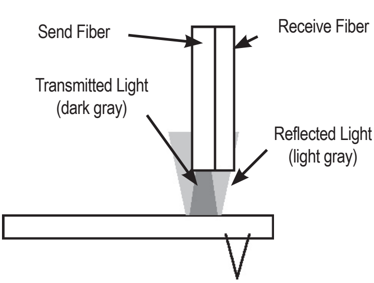
Interferometer:
A Michelson interferometer can be adapted to measure the deflection of a cantilever in an AFM. Although very sensitive, the interferometer was not successful because of fringe hopping. That is, the probe could jump between interference fringes while scanning.



Crystal Oscillator: A piezoelectric crystal such as quartz can be used to measure the force between a probe and a surface. If the probe mounted on the crystal is vibrated and positioned close to a surface, the interaction of the probe and surface will cause a change in the vibration. This change is proportional to force.



Piezo-resistive Cantilevers:
A cantilever can be fabricated that has a small piezo-resitive element in it that changes resistance if the cantilever bends. This type of sensor is viable, but very difficult to manufacture in appropriate quantities.



Fiber Guide:
Light may be transmitted down a fiber optic and reflected off a cantilever into another fiber optic. If the surface is close enough to the fiber optics, then the amount of light that is collected by the second fiber optic depends on the distance from the fiber optic to the cantilever.

FIGURE 2-16 Many measurement methods were tested for measuring the deflection of a cantilever in an AFM.

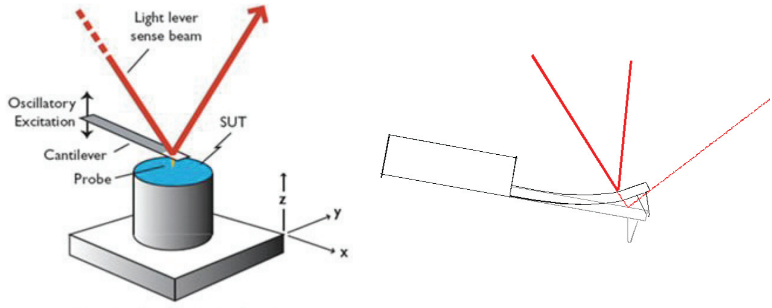


FIGURE 2-17 Left: Illustration of the light lever AFM force sensor. In the LL-AFM sensor the end of the cantilever bends and thus the angle between the probe and sample changes as the cantilever bends. Right: It is possible to fabricate cantilevers with force constants as small as 0.1 nN/nm.

Light Lever Sensors

The design for a LL-AFM sensor is illustrated in Figure 2-17. A laser light is reflected of the back side of a cantilever into a 4 section photo-detector. If a probe, mounted on the backside of the cantilever, interacts with the surface the reflected light path will change. The force is then measured by monitoring the change in light entering the 4 quadrant photo-detector.

As shown in Figure 2-17, the light reflects off the parabolic end of the cantilever, which gives much of the amplification of the light path in the light lever. Geometrically, it is calculated that the deflection at the end of the cantilever is equal to the motion of the laser beam across the face of the photo detector.

The cantilever in the LL-AFM is typically fabricated with a MEMs process. The cantilevers are small, between 50 and 300 microns long, 20 to 60 microns wide, and between .2 and 1 micron thick. Section 2.5 gives a much greater discussion of the cantilever/probes used in an AFM.

The LL-AFM force sensor requires alignment each time a probe/cantilever is exchanged. Typically, alignment is accomplished by first positioning the laser light onto the cantilever, and then confirming that the light is reflected onto the photo-detector by looking at the photo-detectors electronic output. Figure 2-17 illustrates the laser light as it is reflected on the back side of an AFM cantilever.

In the ideal LL-AFM design, the probe would have a 90 degree angle with respect to the surface. Practically, however, this is not possible because of the constraint of the mechanism that holds the probe in place requires that there be an angle between the probe/cantilever and the surface. This angle is usually be between 5 and 15 degrees. Such angles cause artifacts in the images.

Section 2.4 provides a detailed explanation of the modes of operation of the light lever AFM for measuring topography as well as measuring surface physical properties.

Crystal Force Sensors

A force sensor for an AFM may be constructed from quartz crystals. Such sensors are advantageous because they do not require alignment as the LL-AFM force sensor does. The most common types of force sensors are derived from resonating crystals such as a tuning fork or longitudinal quartz resonance oscillator.

Figure 2-18 illustrates a design for a sensor using a longitudinal quartz oscillator. The vibrating motion in this sensor is horizontal to the surface and it works by monitoring the sheer forces between the probe and surface. The motions of the oscillator are less than 0.4 nm and the forces that the sensor can measure are less than 400 pN.

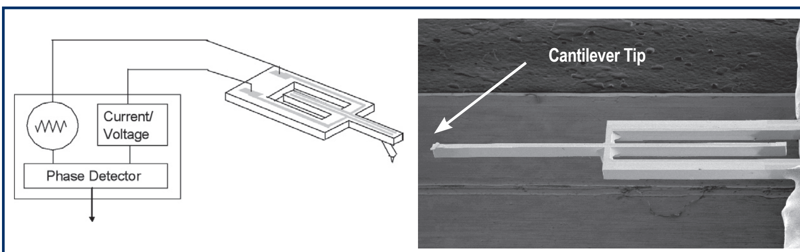


FIGURE 2-18 Left: Crystal force sensor and electronics. Right: SEM image of a crystal force sensor. The cantilever is visible at the left end of the crystal tine.

2.2.3 Integrating LL-Force Sensors and Scanners

The initial AFM designs scanned the sample and kept the probe stationary. This simpler type of design was optimal for only limited types of sample. It became advantageous to design AFM scanners where the X-Y-Z scanner is integrated with the LL-AFM force sensor.

The easiest approach to integrating an X-Y-Z scanner would be to mount the LL sensor at the end of the scanner. This is not feasible because the Z piezo is not responsive enough to move the entire light lever scanner up and down as the probe is scanned across the surface. Such an AFM would be too slow to be practical. Two methods are employed for creating a combined LL-AFM scanner with an X-Y-Z scanner.

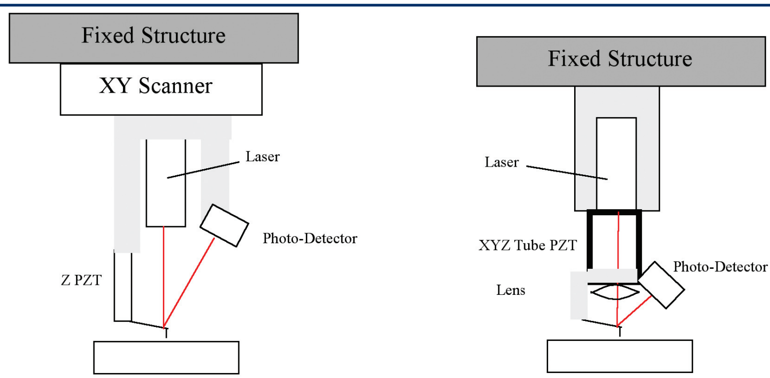


FIGURE 2-19 Two designs for tip scanning light lever AFM scanners. Left: The laser is scanned with the cantilever. Right: The laser is fixed and the cantilever is scanned, a lens keeps the laser light focused on the cantilever.

In the first, illustrated schematically in Figure 2-19, the laser and photo-detector are scanned in the X-Y axis, and the probe is mounted at the end of the Z ceramic. In this design the Z ceramic is part of the light lever optics. That is, as the probe is moved up and down in the Z direction the light path changes. It can be shown geometrically that the Z motion of the cantilever has a minimal effect on the operation of the AFM LL-AFM sensor.

Also illustrated in Figure 2-19, is the other approach commonly used. The laser is held fixed and a lens is used to focus the laser light onto the cantilever. As the lens moves back and forth in the X-Y plane, the laser light stays focused on the cantilever. The photo-detector is then mounted on the X-Y translator.

2.2.4 Z Motors-Probe Approach

One of the major challenges in an AFM is making a motion control system that allows moving the probe to the surface before scanning. This must be done such that the probe does not crash into the surface and break. An analogous engineering challenge would be to fly from the earth to the moon in 60 seconds and stop 38 meters from the surface without overshooting or crashing.

In the AFM stage there are two separate motion generation mechanisms in the Z axis. The first is a stepper-motor-driven mechanism with a dynamic range of a centimeter and a resolution of a few microns. The stepper motor is driving either a linear bearing or an 80 TPI screw. The second motion generation mechanism in the Z axis is the piezoelectric ceramic in the AFM scanner. The z ceramic typically has a dynamic range of about 10 microns or less and a resolution of less than 0.5 nm.

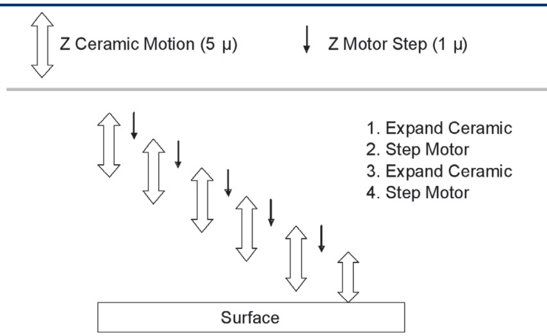


FIGURE 2-20 Motion of probe in the "woodpecker" method for probe approach in an atomic force microscope. The z PZT and Z motor are activated sequentially until the surface is detected.

Typically, probe approach is achieved with a "woodpecker" method, (Figure 2-20). In this method, the stepper motor is stepped a small increment, say 1 micron. Then the z piezoelectric ceramic is extended 5 microns to see if the surface is detected. The z piezo is then retracted, the stepper motor engaged.......on and on. A key component here is that when the probe encounters the surface, the feedback is turned on immediately.

There are two primary mechanisms that may be used for the Z motion control, Figure 2-21. In the first, three lead screws are used together with a kinematic mount. All three screws can be turned simultaneously or a single screw may be turned. If only one of the screws is turned, there is a reduction of motion at the center of the three screws. This geometric reduction in motion can be used to get very precise motion. For automated tip approaches, one of the lead screws may be attached to a motor. In the second method, a linear bearing is used to drive the AFM scanner towards the sample. The linear bearing must be very rigid to avoid unwanted vibrations.

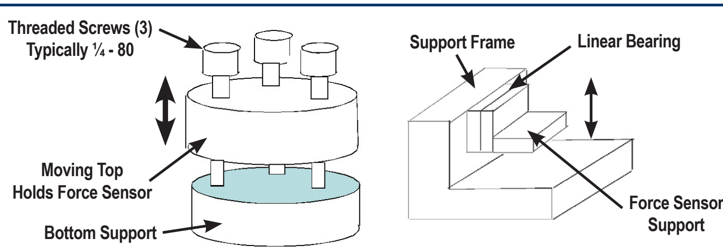


FIGURE 2-21 Left: On AFM's designed for small samples, a kinematic mount is typically used. One of the threaded screws can be motorized for an automated probe approach. Right: A linear bearing can be employed to move the AFM head in the Z axis.

2.2.5 X-Y Stage

Most AFMs include an X-Y position stage for moving the sample relative to the probe. The stage may be manual or automated with motors. The primary function of the X-Y stage is for locating features on a surface for scanning with the AFM. The resolution of the X-Y stage is usually less than 1/10 the range of the x-y scanner that moves the probe.

There are two possible configurations for the X-Y stage. In the first, the sample sits on top of an x and y crossed roller bearing. In the second, the sample is mounted to a block that is directly on the base of the microscope. Typically the base is made from granite. The metal "puck" is then pushed around with the X-Y motors. The advantage of the second design is that there is less chance of the X-Y stage introducing noise into the AFM mechanical loop (see Figure 2-22).

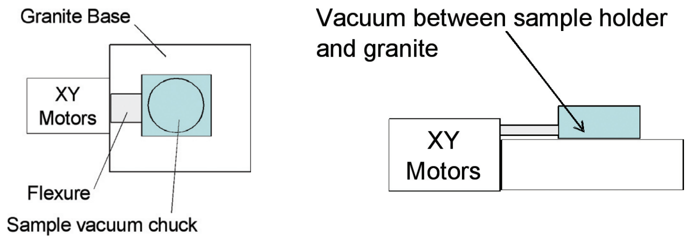


FIGURE 2-22 Configuration for X-Y sample translation stage that gives very low vibration noise. The sample "chuck" is vacuum sealed to the granite base during AFM scanning.

2.2.6 Optic Microscope

The XY stage and optic are not essential for an AFM stage. The optic is used for finding the region for scanning. Also, the optic can be helpful in positioning the laser light on the cantilever in the LL-AFM force sensor. The optical microscope in an AFM can be helpful for probe approach.

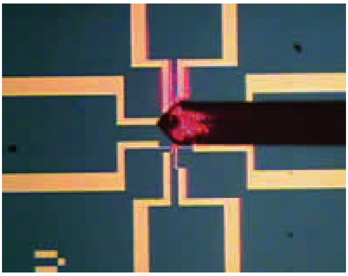


FIGURE 2-23 Video camera image captured of the cantilever/sample in an AFM. The red "spot" is from the laser that is used in the light lever force sensor. Upon scanning ranges greater than 1 micron, it is possible to see the AFM cantilever move in the video microscope image.

There are three optical microscope viewing designs that may be used in an AFM stage, illustrated in Figure 2-24. The 90 degree top down design is optimal for applications when extremely high resolution optical microscope imaging is mandatory. The 45 degree design is helpful for probe approach and is used when high resolution optical imaging is not required. The 90 degree bottom view design is typically used with an inverted optical microscope for biological applications.

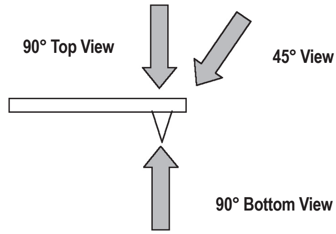


FIGURE 2-24 Illustration of the three viewing positions of an optical microscope in an AFM.

2.2.7 Mechanical Loop

The greatest factor that effects the vertical resolution, or noise floor of an AFM is the rigidity of the mechanical loop. The mechanical loop is comprised of all the mechanical elements between the sample surface and the probe, as illustrated in Figure 2-25. If this loop is not rigid, then the probe can vibrate out of phase from the sample, and noise is introduced into images. It is typically easier to make the mechanical loop very rigid by making the microscope very small. Because of this, in practice the highest resolution AFMs are very small. It is also very difficult to make AFM stages for larger samples such as wafers and discs that have very high vertical resolutions.

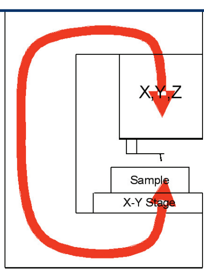


FIGURE 2-25 The mechanical loop in an AFM includes all of the structural elements that are required to hold the probe at a fixed distance from the sample. This includes:

- xyz Scanner
- X-Y Stage
- Z Motor
- AFM Sensor

2.3 Electronics

Most of the electronics in an AFM are resident in a separate cabinet from the stage and the computer. The functions in the electronic controller may be constructed with a DSP chip or analog electronics. This section does not discuss the implementation, but describes the block functions in the controller. The primary function of the electronics in an AFM are to:

a) Generate scanning signals for the x-y piezo

b) Take an input signal from the force sensor and then generate control voltage for the Z piezo

c) Output control signals for X-Y-Z stepper motors

d) Generate signals for vibrating the probe and measuring phase or amplitude when vibrating mode is used for scanning

e) Collect signals for display by the computer

The following sections are a detailed description of these functions.

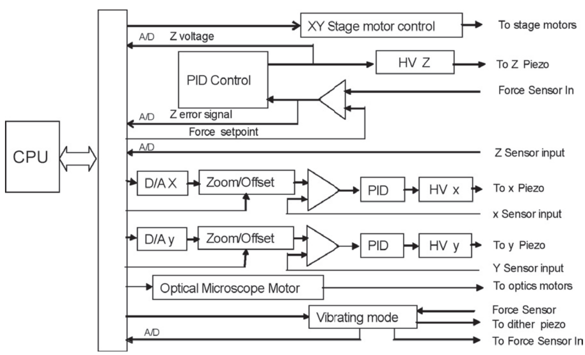


FIGURE 2-26 Block diagram of the functions in an AFM electronic controller as implemented with analog electronics.

As mentioned above, these functions may be implemented with either digital or analog electronics. In the digital approach, see Figure 2-27, all signals from the stage are digitized, and a DSP chip takes care of all of the feedback control calculations. Also, the DSP chip generates the x-y raster scan functions. The advantage of analog electronics is that they are typically less noisy. Because the functionality of a DSP chip is created by a software program, the DSP approach gives a little more flexibility and can be changed very rapidly.

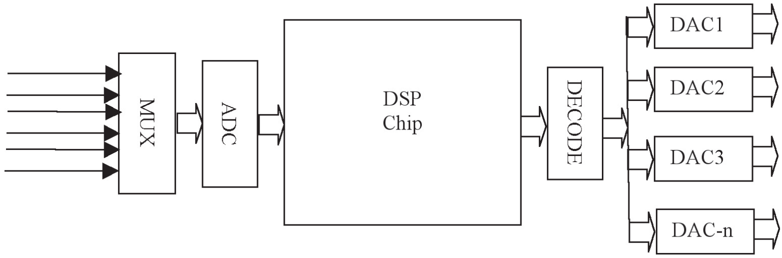


FIGURE 2-27 Block diagram of an AFM electronic controller based on a high speed DSP chip. All of the functions of the analog controller are handled with software in the DSP chip. Inputs to the ADC include the Z signals. Output to the DAC include the signals for the x, y and z piezoelectric ceramics.

XY Signal Generation

The X-Y signal generators create a series of voltage ramps that drive the x and y piezoelectric ceramics in the AFM, as illustrated in Figure 2-28. The scan range is established by adjusting the min and max voltage. The position of the scan is established by offsetting the voltages to the ceramic. Finally, the scan orientation is rotated by changing the phase between the signals.

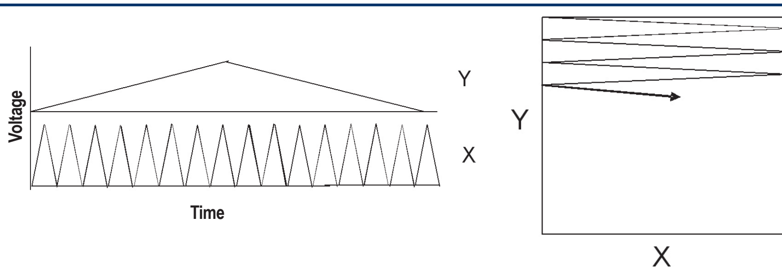


FIGURE 2-28 Left: Signals output for driving the x and y piezoelectric ceramics in the AFM scanner. Right: Motion of the probe in the x and y axis when the piezoelectric ceramics are activated.

A maximum scan range of the AFM scanner is established by the mechanical electrical gain of the ceramics and the maximum voltage the ceramics can tolerate before depolarizing. As an example, ceramics may have a gain of 1 um per volt. If the maximum potential is 100 volts, then the scan range is 100 microns. The maximum achievable resolution is set by the noise floor of the driving voltage. A noise floor of 1 millivolt would give an XY resolution of 1 nanometer.

Equation 2-5: $XY_{range} = G \times PS_v$

Where: XY_{range} = Range of the XY Scanner
PS_v = Power supply voltage
G = Piezoelectric ceramic gain

Equation 2-6: $XY_{anoise} = G \times PS_{noise}$

Where: XY_{anoise} = Analog noise in the XY axis
PS_{noise} = Power supply noise
G = Piezoelectric ceramic gain

It is important that the bit noise associated with the X-Y scan generators be less than the analog noise floor of the electronic controller.

Equation 2-7: $XY_{dnoise} = \frac{XY_{range}}{Bits_{xy}}$

Where: XY_{dnoise} = Digital noise in the XY axis
$Bits_{xy}$ = Number of scanning bits in the XY axis

and

Equation 2-8: $XY_{dnoise} \leq XY_{anoise}$

For example, if the scan range is 100 um and the analog noise floor is less 1 nm, then the number of bits required is at least 100,000, or greater than 2^{17} bits. One option is to use a scale and offset DAC and amplifier if the scan DAC does not give enough bit resolution (see Figure 2-29). As an alternative, a DAC with a much higher number of bits may be used.

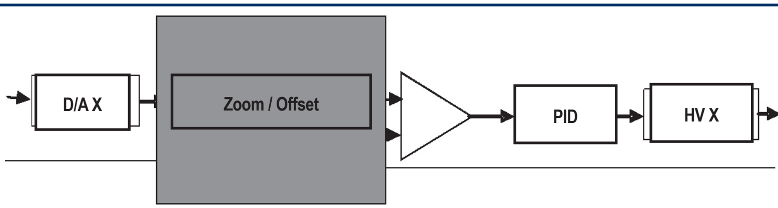


FIGURE 2-29 Block diagram illustration of the placement of zoom/offset electronics in the x or y control electronics. The zoom and offset electronic functions are often implemented with a variable gain and summing operational amplifier.

With digital electronics it is possible to create x and y electronic signals that raster the probe over a specified region of a surface. In general it is best if the drive signals do not have sharp edges at the turning point. Sharp edges can excite resonances in the piezoelectric ceramics, and cause them to vibrate. Such vibrations create unwanted artifacts and "ringing" in the images. Higher speed scanning with an AFM is almost always done using rounded signals such as sinc waves to drive the piezoelectric ceramics.

Feedback Control Circuit

In the AFM, the feedback control electronics take an input from the force sensor and compare the signal to a set-point value; the error signal is then sent through a feedback controller. The output of the feedback controller then drives the Z piezoelectric ceramic.

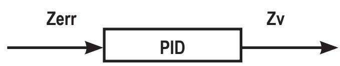


FIGURE 2-30 The PID controller takes the Zerr signal from the force sensor and outputs the Zv signal that drives the Z PZT.

The proportional, integral, derivative controller takes the error signal and processes it as follows:

Equation 2-9: $$Z_v = P \times Z_{err} + I \times \int Z_{err}\, dt + D \times \frac{dZ_{err}}{dt}$$

By selecting the appropriate P, I and D terms in Equation 2-9, the probe will "track" the surface as it is scanned. The integral term facilitates the probe moving over large surface features and the P and D terms allow the probe to follow the smaller, high frequency features on a surface. Two signals may be digitized in the feedback loop to create an AFM image, the error signal, and the Z voltage. When the PID parameters are optimized, the error signal image will be minimal. Section 3.4 describes the process for optimizing the PID parameters in an AFM.

Implementation of the Z feedback loop in an AFM can be made with either analog or digital electronics. The advantage of digital electronics is that they are very flexible and can be configured to do many types of functions. Analog electronics, typically have less noise and have a larger dynamic range. Either approach will typically provide adequate results.

Output of Signals for Stepper Motors

Often an AFM stage has several stepper motors that must be electronically controlled. The stepper motors are typically driven with a series of voltage pulses that are in a specific phase sequence.

The functions in the stage that may be controlled with stepper motors include:

- X-Y sample translation
- Z motion control (1 to 3 motors)
- Zoom/Focus on video microscope

Vibrating Signals

It is often advantageous to mechanically modulate, or vibrate the cantilever in an AFM and to compare the modulated signal phase or amplitude to the drive frequency. Sections 4.3.1 and 4.3.2 provide a detailed explanation of why this should be done. Feedback control may be implemented such that the phase change or the amplitude change is kept constant during scanning.

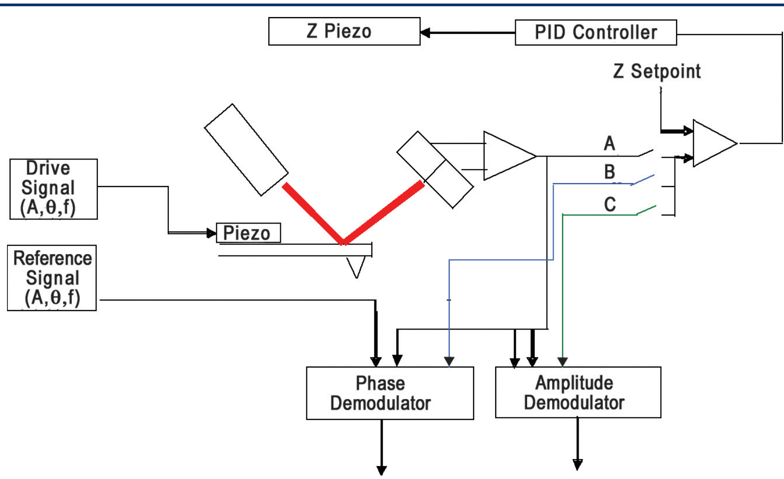


FIGURE 2-31 Block diagram of the electronics employed for vibrating mode AFM scanning. The signal used for feedback can be selected by switch a, b or c. Switch a is for DC feedback, b for phase feedback, and c for amplitude feedback.

Figure 2-31 illustrates the circuit used for mechanical modulation and phase/amplitude detection in the AFM. If the feedback control maintains a constant phase change, then the amplitude may vary while scanning. Vice versa, if the amplitude is maintained constant, then the phase may vary while scanning. For this reason, there are A/D converters to capture and display the amplitude and phase signal.

Collecting Signals

Many electronic signals associated with the Z axis in the AFM are digitized and may be displayed by the master computer. These signals include:

- Z voltage – Voltage that goes to the z piezoelectric ceramic, after the PID controller.
- Z error signal – This signal is proportional to the output of the light lever photo-detector.
- Z Sensor – This signal, from the motion sensor, measures the displacement of the z ceramic in the AFM scanner.
- Amplitude Demodulation – Signal from the amplitude demodulator.
- Phase Demodulation – Signal from the phase demodulator.

In an AFM there are typically one or more high speed digital to analog converters (ADC). If there is a single ADC, the many analog signals are passed through a multiplexer into the ADC input (see Figure 2-32). The speed of the AD converter must be large enough such that at least one data point is converted per pixel.

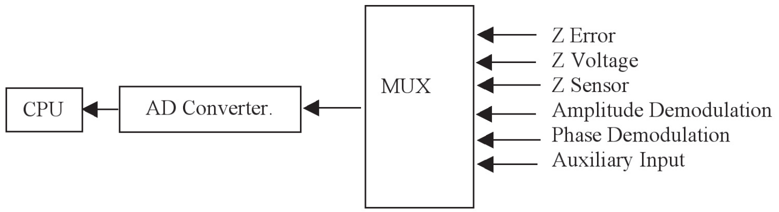


FIGURE 2-32 Several signals are input to the CPU with a single ADC. A multiplexer(MUX) is selects the specific analog input for the CPU.

2.4 AFM Acquisition Software

Typically, a software interface is used for controlling the AFM stage. Functions controlled by software include setting all movement of the X-Y stage to locate the feature for scanning, probe approach to get the probe near/on the surface, setting scan parameters, display of images while scanning, and a capability for measuring F/D curves. Figure 2-33 illustrates a typical AFM scan control window. Below is a list of the functions found in the control software.

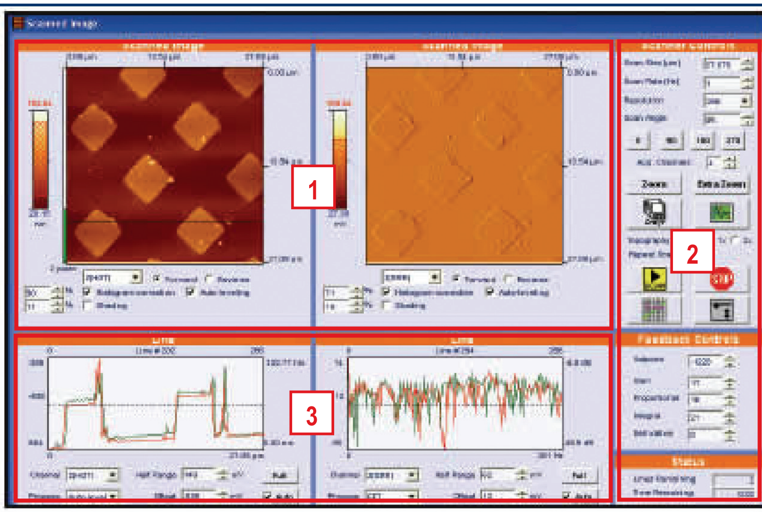


FIGURE 2-33 A window such is this is typically used for acquiring AFM images. There is a section for: (1) displaying images; (2) entering scan parameters; and (3) displaying 2-D profiles.

Display

Visualizing the AFM image in real time is critical to the efficiency of the AFM. This allows an operator to assure that they are scanning the correct region of a sample, and facilitates optimizing the scan parameters such as scan rate and PID settings. Typically there are at least two types of display.

a) A 2-D representation of the image which shows the topography of the specimen being scanned. To correct for tilt between the probe and sample, the image is line leveled (see Section 5.1.1. Without real time line leveling, the image will only show the tilt between the probe and sample.

b) An “oscilloscope window” displays a two dimensional scan line such as the Z signal versus the X axis motion. An oscilloscope window is very helpful for optimizing the scan parameters and assuring that the probe is tracking the sample’s surface.

c) As discussed in Section 2.3, there are a number of data channels that may be monitored in the AFM instrument. They include the Z piezo voltage, Z error signal, Z motion sensor, and phase and amplitude signals. The AFM control software will allow one or more of these signals to be displayed on the screen.

Stage Control

Simplifying the operation of an AFM requires motion control including at least one stepper motor to move the probe relative to the sample in the Z axis. Additional motion control is used for moving the sample in the XY axis relative to the probe as well as controlling the zoom and focus of an optical microscope.

a) Z motion control: Probe approach is a very important function in the AFM (see Section 3.3). The Z approach software should be rapid, but it should not allow the probe to touch the surface such that the probe breaks. Properly optimized, probe approach takes less than a minute.

The approach software typically has several options for controlling the rate at which the probe moves toward the sample’s surface. Software algorithms are also critical for setting the threshold signal levels associated with the probe interacting with the surface. Once the threshold is met, the approach is stopped and the AFM is put into feedback.

If there is an automated video microscope, the software algorithm for tip approach can be augmented to shorten the time required for probe approach. This is achieved by focusing the microscope on the probe, then the sample. The relative positions of the probe and sample are compared. Then the Z motors are driven rapidly until the probe is less than 100 microns from the surface.

b) XY motion control: Because the XY motion in the AFM is typically less than 100 microns, an XY motion control system is required that facilitates moving the probe to within a few microns of the features that are to be scanned. An XY positioning table driven with stepper motors is often used. Software is then used to move the translation stage. The software typically is activated by a track ball or by curser activation of an icon in a window.

Advanced software functions may be added to microscopes with automated X-Y stages. Functions include an ability to measure many images adjacent to each other, and to measure several images on pre-set locations on the sample. It is possible to drive the stage to pre-established locations for inspection applications with registration software.

X-Y Scan Control

The exact scan control parameters that are used for scanning a sample depend on the particular application. There are a few variables that must be selected to scan a sample. They are:

a) Image Size: This is the window that is selected for viewing the features on a surface. The image size should be at least as large as the features that are to be visualized. Often a large scan is measured and then the operator "zooms" in on a feature of interest.

b) Number of lines in the image: The digital resolution of the image is established with the number of lines selected for the image. For example, if the scan size is 10 X 10 microns and the number of lines selected is 256 then the digital resolution is 39 nanometers. The number of lines in the image may range from less than one hundred to several thousand.

c) Image rotation angle: The image scan angle may be changed with software. Rotation angles between 0 and 360 degrees may typically be selected. Rotating the image scan axis often means that the largest scan range can not be achieved.

Z Control

Software is required for controlling the feedback control electronics, see Section 2.3. There are two functions that are controlled; the set point voltage and the PID parameters.

a) Set-point voltage: This is the voltage that goes into the differential amplifier, so this voltage is compared with the force sensor output voltage and an error signal is generated. The set point voltage controls the "relative" force. A calibration of the specific cantilever is required to convert the set-point voltage to a force (see Section 2.5).

b) PID parameters: These parameters control the "responsiveness" of the feedback control electronics. These parameters must be adjusted such that the probe tracks the surface while scanning. Section 3.4 provides a description of optimizing the feedback control parameters.

F/D Curves

Force distance (F/D) curves are used to measure the forces experienced by the probe as a function of distance from the surface. In F/D measurements, the probe is moved toward the sample surface to a pre-selected position, and then retracted. The extent of cantilever deflection over the course of this movement is expressed by the Z(ERR) signal which is used to generate a force-distance curve. Software for making F/D curves, illustrated in Figure 2-34, has several variable parameters including:

a) Start and end position for probe

b) Rate of probe approach motion

c) Number of F/D curves to signal average

d) Location on image for F/D curve

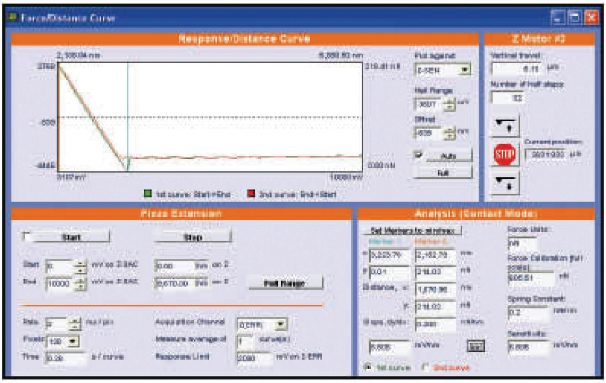


FIGURE 2-34 Software window used for measuring force/distance curves with an AFM.

2.5 LL-AFM Cantilevers and Probes

An LL-AFM force sensor requires a cantilever with a probe at its end for operation. Typically these are fabricated using MEMS technology and are considered a disposable component of the AFM. In principle, an AFM probe should last forever; however, in practice the probe tip is often blunted when it touches a surface. Changing the probe typically takes only a few minutes. Figure 2-35 illustrates the geometry of a typical probe/cantilever/substrate.

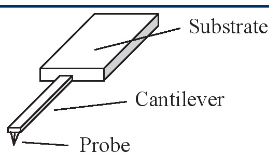


FIGURE 2-35 Illustration of an AFM cantilever/probe/substrate created by micromachining of Si or SiN. All commercially available probes have substrates with the same dimension.

The geometry of the probe is critical to the quality of images measured with an AFM. All AFM images are a convolution of probe geometry and surface. As an example, in Figure 2-36, if the probe cannot reach the bottom of the surface feature, the image will not indicate the correct geometry of the sample.

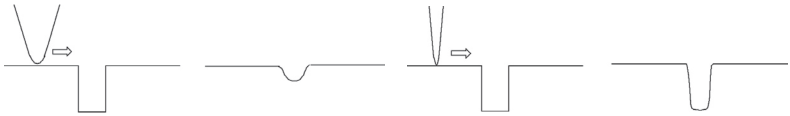


FIGURE 2-36 Comparison of line profiles measured with a dull probe (Left) and a sharp probe (Right). The profile made with the sharp probe shows the bottom of the trench, whereas the dull probe cannot reach the bottom of the trench. The sharp probe is unable to reach the bottom corners of the trench.

Materials

In principle, AFM cantilevers can be fabricated from any material that can be fabricated into a spring-like cantilever. The first AFM cantilevers were fabricated from tungsten wire and had a probe etched in the silicon at the end. Early in the evolution of AFM it was discovered that the best AFM probes could be constructed from MEMs technology. There are two materials commonly used for AFM cantilevers: SiN and Si.

SiN is used for creating probes that have very low force constants. The thin films used for creating SiN probes must have very low stress so the cantilevers don't bend naturally from the stress. Practically, most SiN films have some residual stress and in fact, cantilevers made with SiN tend to have curvature along their primary axis.

Cantilevers fabricated from silicon tend to have less residual stress than SiN and tend not to suffer from bending. However, the Si probes that are fabricated at the end of the cantilever can be brittle and tend to chip if they contact a surface. Most of the cantilever/probes used in LL-AFM force sensors are constructed from Si.

Basic Geometry of Cantilever and Probes

Two basic geometries are used for AFM cantilevers, rectangular and triangular. The two primary shapes for probes are pyramidal and conical. Typically SiN probes are pyramidal and Si probes are conical, see Figure 2-37.

AFM cantilevers were initially fabricated from SiN in a triangular shape. Because of the cantilevers bending, Si became the preferred material.

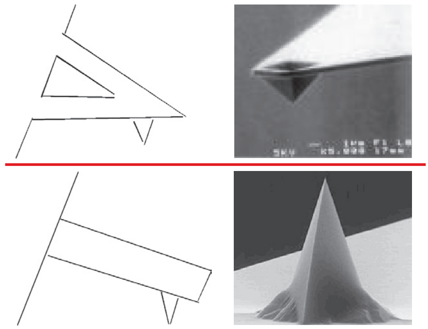


FIGURE 2-37 SiN cantilevers are typically triangular with two arms meeting at an apex. The probe on SiN probes are typically pyramidal and appear hollow at the top. (Top) Si cantilevers are typically rectangular and the probes tend to have a triangular shape to them. Si probes are crystalline and are prone to chipping and breaking if they crash into a surface. (Bottom)

Contact Versus Non-Contact

LL-AFM force sensors can be operated in two basic topography modes; contact mode and vibrating mode, see Section 4.1. The cantilevers used for contact mode have force constants that are typically less than 1 N/m and are fabricated from Si and SiN. On the other hand, vibrating mode cantilevers are typically fabricated from Si and have force constants that are greater than 10 N/m.

Equations for Cantilevers

The rectangular cantilevers used in LL-AFM force sensors have the same mechanical properties as all cantilevered beams. They have a vertical force constant and resonant frequency given by Equation 2-10. Additionally a cantilever has torsional and bending force constants given by Equations 2-11 and 2-12.

Equation 2-10: $$k_{ver} = w \times \frac{E}{4} \times \left(\frac{t}{l}\right)^3$$

Equation 2-11: $$k_{lat} = t \times \frac{E}{4} \times \left(\frac{w}{l}\right)^3$$

Equation 2-12: $$k_{tor} = w \times \frac{G}{3} \times \frac{t^3}{l} \times \frac{1}{\left(H + \frac{t}{2}\right)^2}$$

Where:
k_{ver} = Vertical force constant
k_{tor} = Torsional force constant
k_{lat} = Lateral force constant
E = Young's modulus
G = Modulus of rigidity
w = Cantilever width
l = Cantilever length
t = Cantilever thickness

Specialty Coatings/Configurations

As detailed in Chapter 4, the AFM can make many other measurements besides topography measurements. Many of these measurements require probes with specialty coatings such as platinum or cobalt. Typically these coatings are used only on Si probes because Si probes do not bend as much as SiN probes when coated.

One method for controlling the geometry of the probe on an AFM cantilever is to mount a sphere at the end of the cantilever. The sphere can be mounted directly on a cantilever that does not have a probe. The sphere may also be mounted at the end of a "plateau probe", or a probe that does not have sharp tip, but instead has a flat "plateau" (see Figure 2-38).

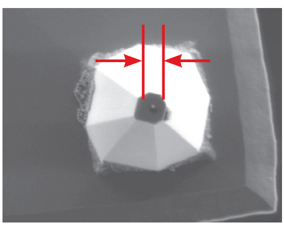


FIGURE 2-38 This silicon probe has a 300 nm latex sphere glued to its apex.

Sharpened Probes

Horizontal resolution with an AFM is improved with sharper probes. There are many techniques available for sharpening AFM probes.

a) SiN probes are sharpened by adding an extra process step that changes the shape of the pit that the SiN film is deposited on. However, this technique often gives double tips which can cause substantial artifacts in images.

b) Si probes can be sharpened by chemical etching, ion milling or by adding a carbon nanotube (see Figure 2-39). Each of these techniques can create a sharper probe, but add a lot of cost to the price of fabricating the probe.

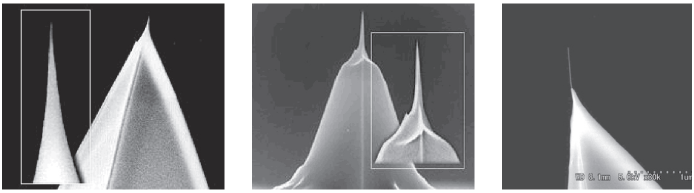


FIGURE 2-39 SEM images of 3 types of sharpened silicon probes. Left: probe sharpened with electrochemical etch. Center: probe sharpened with ion milling. Right: probe sharpened with carbon nanotube.

Probe Cantilever Errors

Probe/Cantilevers fabricated with MEMS processing technologies are subject to fairly dramatic distributions of specifications. For example, a wafer of probes will have only 85% of the probes less than 10 nm in diameter. The other 15 % could have any diameter. This distribution can dramatically effect the quality and resolution of AFM images.

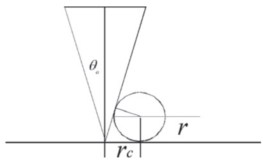
Besides the probe geometry, the cantilevers can have a substantial variation in specifications, especially the thickness of the cantilevers. Because the force constant of the cantilever varies as the inverse cube of the thickness, the force constants of MEMS fabricated cantilevers can vary dramatically. Figure 2-40 is an illustration of the variability and cantilever geometries and the calculated impact on critical specifications.

Cantilever Data	Value	Range
Thickness	2 µm	1.5 - 2.5
Mean Width	50µm	45 - 55
Length	450 µm	445 - 455
Force Constant	0.2 N/m	0.07 - 0.4
Resonance Frequency	13 kHz	9 - 17

FIGURE 2-40 Nominal values for geometric parameters in AFM cantilevers.

Probe De-Convolution

If the probe geometry is analytically known, it is possible to remove the probe geometry's effect on an AFM image (see Figure 2-41). Unfortunately the geometry of AFM probes is not well known, and this approach for removing probe geometry from images is not practical.



$$r_c = r\left(\cos\theta_o + \sqrt{\cos^2\theta_o + (1+\sin\theta_o)\left(-1+\left(\frac{\tan\theta_o}{\cos\theta_o}\right)+\tan^2\theta_o\right)}\right)$$

FIGURE 2-41 If the probe geometry is described as an upside down triangle, it is simple to remove the effect of the probe from an image of a nanoparticle. The actual diameter of the nanoparticle is calculated using the equation at the right. Rarely is a probe in the shape of an upside down triangle.

However, using a technique called blind reconstruction, the geometry of a probe can be calculated by carefully analyzing an image. Blind reconstruction works by searching for repeated patterns in an image and using them to figure out the probe geometry. It is possible to mathematically remove the probe broadening artifacts from AFM images after the probe geometry is calculated.

As an example, Figure 2-42 is an AFM image of 100 nm diameter nano-spheres. By using the blind reconstruction technique, it is possible to calculate the probe geometry from one of the isolated nano-spheres. Then the probe geometry can be removed from the entire image.

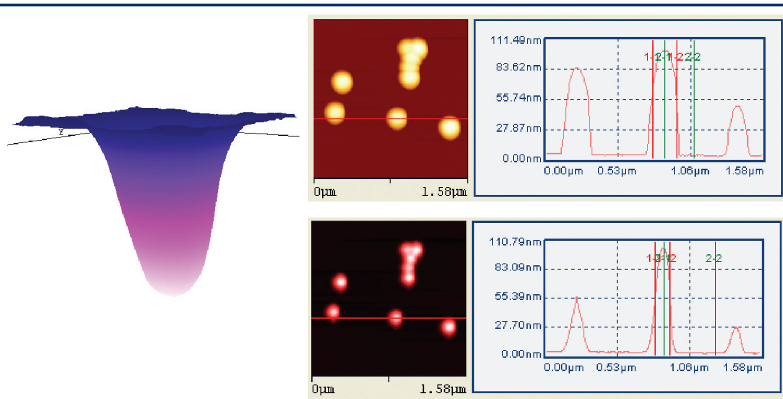


FIGURE 2-42 Blind reconstruction of the nanoparticle image at the top right gives the nanoparticle image at the bottom right and an image of the probe that was used for measuring the original image.

Such methods are very computer intensive and it can take a long time to analyze a single image. For example, to do the blind reconstruction on a single 256 X 256 pixel image with an 3 GHz Pentium PC can take 10 minutes.

Probe Damage

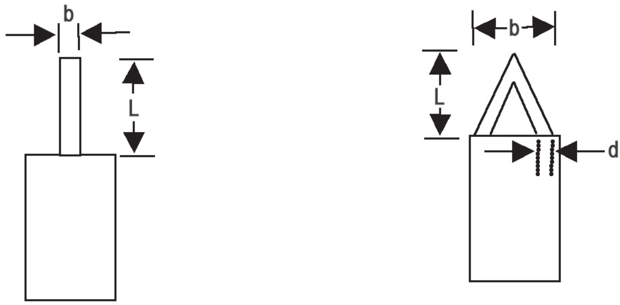
The quality of an AFM image is critically dependent on the shape of the probe used for measuring an image. The AFM probe can be severely damaged by tip approach (see Section 3.3). Handling the probe incorrectly before it is placed in the microscope can also cause probe damage. For example, if the probe is exposed to high electric fields, the probe tip can be blown off by electrostatic discharge. Finally, the probe tip can get dirty from the packing materials used to hold the probe while shipping.

Multiple Probes

The scan rate of an AFM limits the sizes of areas that can be analyzed to a few hundred microns at best. It would be highly desirable to create an AFM with multiple probes that could scan many areas simultaneously. Several efforts to create "multiple probe" atomic force microscopes demonstrated that it is possible. In the first approach, several AFM scanners were positioned above a silicon wafer and scanned independently. In the second approach, several probes on a silicon wafer were used to scan a sample simultaneously. The greatest challenge for creating multiple probe AFM instrumentation is to get all of the probes to be as sharp as is required for high resolution scanning.

Calibration of Cantilever Force Constants

As illustrated in Figure 2-40, there is considerable variability in the force constant of an AFM cantilever because of variations primarily in the thickness of the cantilevers. Thus, if the exact force is required for an AFM scan or F/D curve, the force constant for the cantilever used for the tests must be calculated. The primary method used for doing this is the Sader Method. In this method, the physical geometry of the cantilever is measured with an optical microscope and the quality factor, Q, is measured. These parameters are then used in an equation that calculates the force constant (see Figure 2-43).



Equation 2-13: $k_{rec} = 0.1906\rho b^2 L Q_r \omega_f^2 \Gamma_i^f(\omega_f)$

Equation 2-14: $k_{tri} = \frac{D_0 \bar{d}}{2L^3}\left(1 + 4\frac{\bar{d}^3}{b^3}\right)^{-1}$

Where:
k_{rec} = rectangular force constant
k_{tri} = triangular force constant
ρ = density of fluid(or medium)
Q_r = Quality factor at resonance
ω_f = Resonant frequency
$\Gamma_i^f(\omega_f)$ = imaginary part of hydrodynamic function

$$D_0 = k_{rec}\frac{4L^3}{b}$$

$$\bar{d} = \frac{d}{\sqrt{1 + \frac{b^2}{4L^2}}}$$

FIGURE 2-43 Equations for calculating force constants with the Sader Method.

2.6 Miscellaneous Topics

There are many subjects associated with AFM instrumentation that do not fall under any of the traditional categories. These include the effect of vibrations on an AFM, scanning in controlled environments, and heating and cooling stages.

2.6.1 Vibrations

Vibrations that can degrade the resolution of AFM images can come from two sources:

a) Acoustic Vibrations that are transmitted through the air and have their source from fans, human voices, and other sound sources. Acoustic vibrations can be reduced by putting the AFM in a sealed box that is constructed from acoustic dampening materials.

b) Mechanical Vibrations are transmitted to the AFM stage from mechanical contact. Sources of mechanical noise include floor vibrations. Usually floor vibrations are greater the higher up in a building the AFM is located. Mechanical vibrations are reduced by putting the AFM on a vibration table.

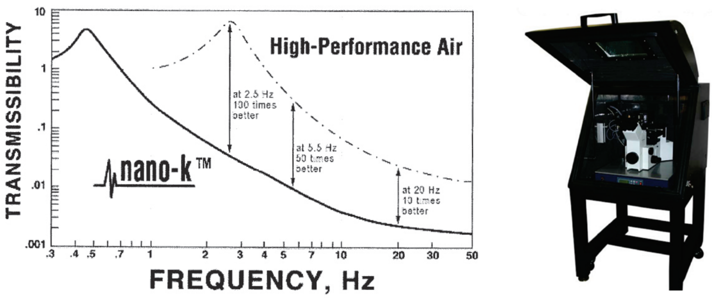


FIGURE 2-44 Left: Vibration tables have a transmission curve that shows the attenuation of the table to vibrations as a function of frequency. There is always a natural resonance associated with the vibration table that is typically below 5 Hz. Right: Acoustic vibrations are reduced using a vibration enclosure.

2.6.2. Environmental Scanning

Scanning in a controlled environment with an AFM can be advantageous for many reasons:

a) Reactive materials are stable in an inert environment.

b) Biological materials can be measured in situ.

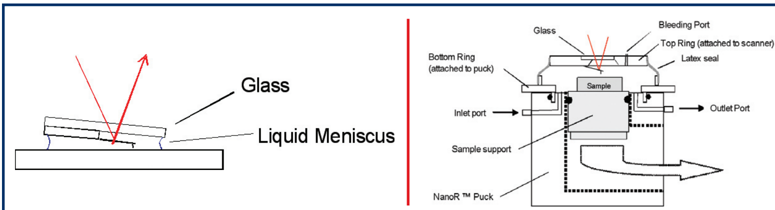


FIGURE 2-45 Left: A microcell is created by gluing the cantilever/substrate on to a piece of glass. Right: An environmental cell is created by making a sealed cell that completely surrounds the probe/cantilever and the sample.

There are many ways for controlling the environment of scanning with an AFM. The first is to place the AFM in a vacuum chamber and remove all of the gasses from the microscope stage. The second is to put the AFM in a controlled environment such as a glove box. The third is to put the sample and AFM tip in a liquid environment. Figure 2-45 shows two methods for putting the sample/probe in a liquid environment.

2.6.3 Heating/Cooling Stages

Often it is desirable to heat and cool samples so that they can be scanned at temperatures other than room temperature. The limitations of the temperatures that may be used are:

a) at the low temperature - condensation on the sample

b) at the high temperatures - destruction of the AFM scanner

The practical temperature range is between –20 C and 200 C.

As a sample is heated or cooled, there will be considerable thermal drift in the AFM stage in the XY and Z directions. The thermal drift can only be minimized by making the stage as symmetrical as possible and using low drift materials. Usually there is drift as the stage temperature is raised, and the drift stops when the desired temperature is reached and the stage temperature is stabilized.

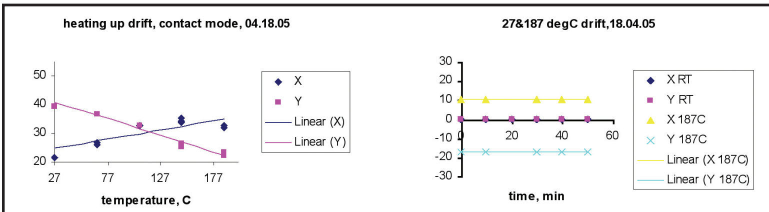


FIGURE 2-46 Left: Scanner drift in the x and y axis as a sample is heated in an AFM sample stage. Right: Scanner drift after the sample reaches a specified temperature in an AFM sample stage.

Cooling samples in an AFM stage has another potential problem. Condensation occurs on the components that are being cooled. When cooling a sample in an AFM stage, it is recommended that the entire stage be maintained in a dry environment such as in a glove box.

2.6.4 Higher Speed AFM Scanning

Typically an AFM scan rate is approximately 1 Hz. A scan with 254 lines will take approximately 4 to 5 minutes. It would be advantageous to have an AFM that scans at much higher scan rates. Constructing a high speed AFM requires creating mechanical control systems that have very high first resonant frequencies. There are two methods used for making an AFM that scans samples faster. They are:

Cantilever Deflection

In this approach, the sample or tip is scanned at a high slew rate with an AFM scanner having a very high first resonant frequency. Feedback is used to follow the general or low frequency surface topography associated with tilt between the scanner and sample. The cantilever then bends up and down as the probe glides across the surface; the probe has a variable force as it moves over the surface. An image is generated by monitoring the up and down motion or deflection of the cantilever. Typically, a cantilever with a relatively small force constant is used so that the probe does not scratch the surface. The "cantilever bending" approach is used for scanning small scan ranges, less than 5 microns, and samples with minimal topographic variations.

Full Feedback

Creating an AFM with full feedback capabilities that scan many types of samples is a formidable challenge. The following criteria must be met:

a) x-y scanner with a very high first resonant frequency

b) force sensing system with extremely high frequency response

c) high bandwidth feedback control electronics

d) Z piezoelectric or other motion control mechanism with a very high first resonant frequency

The scan rate is limited by the criteria a-d that has the slowest responsiveness. For example if there is a system that has a high response sensing, feedback controller, and Z actuator but the x-y scanner has a slow response, then the scan rate of the AFM is established by the x-y scanner.

CHAPTER 3

Measuring AFM Images

Learning to operate an AFM well enough to get an image usually takes a few hours of instruction and practice. It takes 5 to 10 minutes to measure an image if the sample is properly prepared. However, if it is an unknown sample that has never been scanned with an AFM before, it can take substantially more time to acquire meaningful images. The following sections discuss the steps required for measuring an AFM image, illustrated in Figure 3-1.

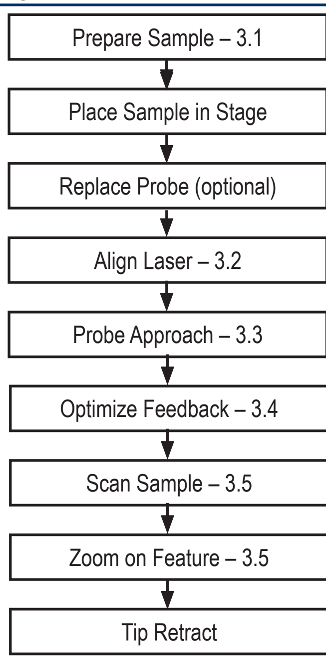


FIGURE 3-1 Sequential steps required for measuring an AFM image.

This chapter assumes that contact mode is being used for scanning. If a vibrating mode is being used, Section 3.2 will change to include measuring the resonant curve of the cantilever.

3.1 Sample Preparation

Sample preparation for an AFM is reasonably simple. There are a few basic rules that must be followed to adequately prepare a sample for AFM scanning. The rules are:

a) Sample must be adhered to the surface: If the sample has material adhered to the surface, the material must be rigidly mounted to the surface. If the material is not rigidly adhered two problems can occur. First, the probe can push the material to the edge of the scan range. When this occurs, the image appears as though there is nothing on the surface and only the substrate is observed. Second, the probe can pick up material from the surface because the material has a greater affinity for the probe than the surface. In this case the images often have streaks in them. The streaks are created by material moving on and off the probe, i.e. the probe geometry is changed by the material from the surface.

b) Sample must be clean: AFM imaging requires that the probe move directly across the sample's surface topography. If the surface is dirty with a thick contamination layer, the probe needs to penetrate through the contamination layer to reach the surface. The contamination layer then causes severe distortion in the image (see Section 6.5.1).

c) Sample dimensions must be realistic: The AFM can image a large variety of samples; however, there are a few constraints. Features on the sample's surface must be smaller than the dynamic range of the Z ceramic. Typically this is less than 10 microns. If the features on the surface are larger than 10 microns, then the Z piezo will not be able to move the probe over the features. Second, the probe must be able to directly access the features. As an example, if the sample has a 10 nm diameter hole, and the probe is 40 nm in diameter, the probe will not reach into the hole.

d) Sample must be rigidly mounted in the AFM stage: When the sample is fastened into the AFM stage, it must be mounted rigidly. If the sample is not mounted rigidly, it can vibrate. Vibrations substantially reduced the resolution of the microscope and often make it impossible to see small surface features.

3.2 Probe Laser Alignment

If there is no probe in the AFM scanner, or the probe in the scanner is broken, a new probe must be inserted. The specifics of inserting a probe into the microscope depend on the particular type of AFM being used. It usually takes only 30-40 seconds to replace the probe. The probe must be selected such that it matches the mode and application. After the probe is securely fastened into the AFM scanner:

a) Adjust laser on cantilever – An AFM scanner has two laser adjustment screws, one for moving the laser in the X direction, and one to move the laser in the Y direction. These screws are adjusted so that the laser light is on the end of the cantilever. If the AFM stage has an optical microscope, the laser can readily be seen on the cantilever.

b) Move detector – Like the laser, the photo detector has two adjustment screws, one for the X and one for the Y direction. The photo-detector position is adjusted so that the laser is at the center of the photo-detector. Typically, a software window has information that helps adjust the detector (see Figure 3-2).

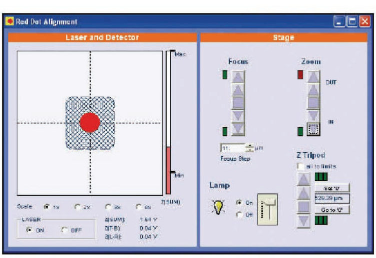


FIGURE 3-2 This software window shows the position of the laser on the photo-detector. By moving the position of the photo-detector in the x-y axis, the position of the red dot moves relative to the photo detector. At the right of the red dot is a vertical red bar that indicates the total laser power on the detector.

3.3 Probe Approach

Once the sample and cantilever are in the microscope stage, the next step is to initiate a probe approach. Probe approach moves the probe from approximately 1 mm from the surface to a condition of feedback. If tip approach is not implemented correctly, there is a great risk that the tip will crash into the surface and break.

Typically, the woodpecker method is used for doing tip approach. In the woodpecker method, the probe is moved in steps in the Z direction towards the surface until the force sensor detects forces associated with the surface. Section 2.3.4 describes the woodpecker method for probe approach. Figure 3-3 illustrates an SEM image of a probe that was damaged in tip approach.

Probes crash into the surface if the probe approach is made too rapidly or if the feedback electronics are not switched on rapidly enough after the surface is detected by the force sensor.

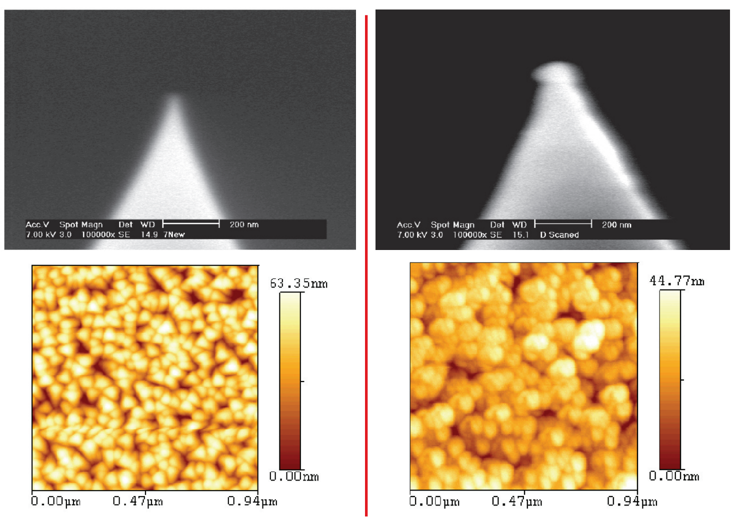


FIGURE 3-3 An operator must know how to operate the AFM such that probes are not damaged. Left: SEM image of sharp probe and an AFM image measured with the sharp probe. Right: SEM image of damaged probe and an AFM image measured with the damaged probe.

3.4 Optimizing Scan Conditions

Assuming that probe approach is completed, the AFM probe can be scanned across the surface. The scanning can be made in two dimensions; the probe is scanned in a line scan, back and forth across the surface. Alternatively, a scan can be initiated. The motion of the probe as well as the Z error signal are displayed in a two dimensional oscilloscope window (see Figure 3-4).

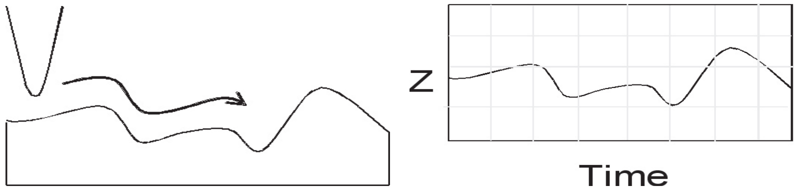


FIGURE 3-4 Optimizing the PID parameters is done by assuring that the probe is tracking surface features. An oscilloscope window is often helpful for this.

The scan parameters such as the set-point voltage, and the PID parameters are adjusted as the line scan is being made. The goal in adjusting the scan parameters is to have the probe track the surface. The probe is tracking the surface when the Z error signal image has a minimal signal. Establishing the optimal conditions requires practice and some intuition.

When first learning to operate an AFM, it is helpful to operate with a test sample and adjust the PID settings to see the effect on the Z voltage and the Z error signal, as shown in Figure 3-5.

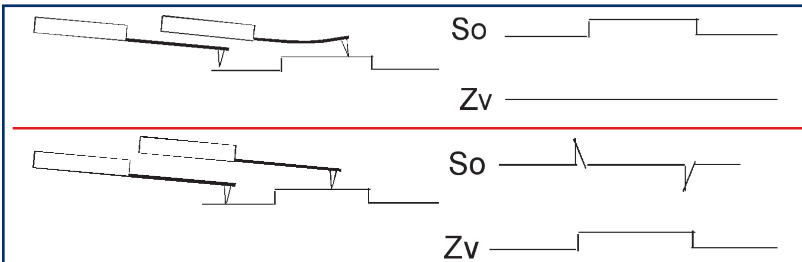


FIGURE 3-5 Top: If the PID parameters are all zero, the cantilever will bend as it moves across the surface features. Bottom: If the PID parameters are optimized, the cantilever deflection remains constant while scanning.

3.5. Scan Image / Zoom

After the scan parameters are optimized, a scan is initiated. The range of the first scan depends on the specific sample being examined. A scan that is far greater than the desired features is typically made. After the initial scan, a zoomed scan is made of the specific region of interest (see Figure 3-6). Often it is necessary to zoom in many times before it is possible to get an image of the region of interest.

After the scanning is completed, the tip retract function is activated. Once the probe is removed from the surface, the sample can be removed from the microscope stage.

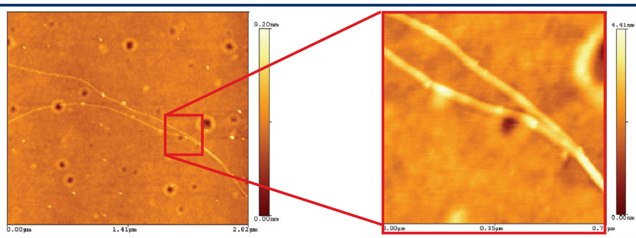


FIGURE 3-6 Typically a large area is scanned (left) and then a smaller area is scanned so that a high resolution image is made of a specific area.

3.6 AFM Scanning Suggestions

High Resolution Scanning

Learning to measure AFM images with a resolution of 50 nm is very simple. It can be considerably harder when higher resolution images are required. It is recommended that when learning to measure images with < 50 nm resolution, a tip check sample is used. After practicing with the tip checker sample and getting great images, switch to other "unknown" samples.

Choosing a Topography Scanning Mode

There are two primary topography scanning modes (see Section 4.1), contact mode and vibrating mode. Contact mode should be used with hard samples and when a resolution of > 50 nm is required. Vibrating mode should be used on soft samples and when a resolution of < 50 nm is required.

False Feedback

Sometimes, especially with vibrating mode, the AFM will enter a "false feedback" condition during probe approach. In the false feedback condition, tip approach is stopped when the probe is slightly above the surface. Often a false feedback is caused by contamination on the surface. In the event of false feedback, the Z motors can usually be overridden to get the probe closer to the surface.

Damaged Probes

The most frequent problem that occurs in an AFM is that the probe is broken before the image is measured. The probe could be broken in tip approach or it could be broken before it is placed into the microscope. It is suggested that, when it is not possible to get a high resolution image, the probe be changed.

CHAPTER 4

Imaging Modes

The Atomic Force Microscope is a member of the family of scanning probe microscopes that includes the scanning tunneling microscope and the near field optical microscope, see Figure 4-1.

Scanning Probe Microscope Family

Air, Liquid, Vacuum

Atomic Force Microscope (AFM)

- Contact Mode
  - Lateral Force (LFM)
  - Scanning Thermal (SThM)
  - Lithography
  - Shark
- Vibrating Mode
  - Close Contact (CC)
  - Intermittent Contact (IC)
  - Magnetic Force (MFM)
  - Electric Force (EFM)
  - Kelvin Probe (SKPM)
- Electrochemistry

Scanning Tunneling Microscope (STM)

- Topography
- Spectroscopy
- Lithography
- EChem.
- BEEM

Near Field Optical Microscope (NSOM)

- Aperture
- Aperture-less
- Reflection
- Transmission

FIGURE 4-1 There are several types of Scanning Probe Microscopes that are used for measuring surface topography and physical properties. The primary types of SPM's are the AFM, STM and NSOM. AFM's account for about 80% of the total number of scanning probe microscopes.

Each of these microscopes measures surface topography by raster scanning a small probe across a surface and monitoring the probes motion. A scanning tunneling microscope (STM)[1] operates by monitoring the current flow between a probe and surface. In the atomic force microscope

(AFM)[2], the force between a probe and surface is monitored. Lastly, in the near field optical microscope (NSOM)[3] the optical properties of a sample's surface are monitored.

Soon after the invention of the AFM it was realized that these instruments were capable of measuring far more than surface topography. In fact, it is possible to measure almost any physically observable phenomena at the nanometer scale. The only requirement is that a nanoscopic sensor must be developed for the end of a probe. For example, magnetic fields, electric fields, temperature, and hardness may be measured with the AFM probe. Additionally, it is possible to use the AFM probe to modify surfaces. By definition, an AFM mode is a non-topographical measurement made with an AFM.

For the most part, atomic force microscopes are operated in ambient air. At the surface of samples maintained in ambient air, there is always a contamination layer comprised of water and hydrocarbons. Thus, in an AFM, the probe tip is typically immersed in the contamination layer (see Figure 4-2). Because the contamination layer can vary from one environment to the next, the layer can cause uncertainty in AFM measurements. This is especially true for mode measurements made with AFM.

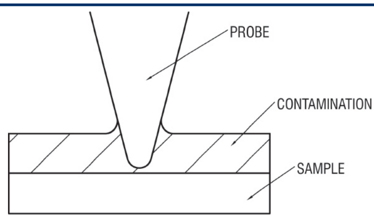


FIGURE 4-2 In ambient air, the AFM probe must pass through a surface contamination layer to touch the surface.

AFM probes contribute a lot of uncertainty of topography and mode measurements. The uncertainty is due to variations in probe geometry. As provided by a vendor[4], the typical AFM probe has a diameter of < 15 nm. That is to say it could be 15 or 5 or 10 nm in diameter. The uncertainty goes up when the probe is coated with a thin film of metal or other type of material. Not only are there variations in the probe coating thickness, there can be variations in the integrity of the probe. For example, the coating on an AFM probe may have grains. Figure 4-3 shows an SEM image of a typical AFM probe and an AFM probe coated with a conductive diamond film.

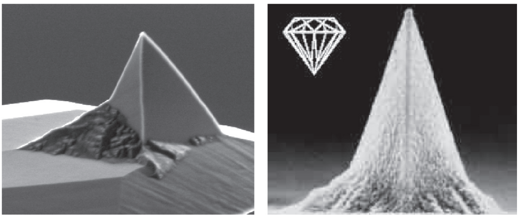


FIGURE 4-3 Left: SEM image of standard AFM Probe. Right: SEM image of an AFM probe coated with a conductive diamond film.

4.1 Topography Modes

A force sensor in an AFM can only work if the probe interacts with the force field associated with a surface. In ambient air, the potential energy between the probe and surface is shown in Figure 4-4. There are three basic regions of interaction between the probe and surface:

- free space
- attractive region
- repulsive region

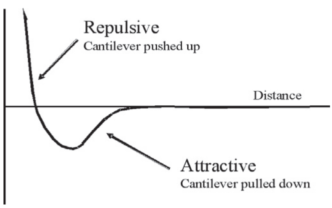


FIGURE 4-4 Potential energy diagram of a probe and sample. The attractive potential is caused by the capillary forces from surface contamination.

Attractive forces near the surface are caused by a nanoscopic layer of contamination that is present on all surfaces in ambient air. The contamination is typically an aerosol composed of water vapor and hydrocarbons. The amount of contamination depends on the environment in which the microscope is being operated. Repulsive forces increase as the probe begins to "contact" the surface. The repulsive forces in the AFM tend to cause the cantilever to bend up.

There are two primary methods for establishing the forces between a probe and a sample when an AFM is operated. In contact mode the deflection of the cantilever is measured, and in vibrating mode the changes in frequency and amplitude are used to measure the force interaction. As a rule of thumb, the forces between the probe and surface are greater with contact modes than with vibrating modes.

4.1.1 Contact Modes

In contact mode, the cantilever is scanned over a surface at a fixed deflection, Figure 4-5. Provided that the PID feedback loop is optimized, a constant force is applied to the surface while scanning. If the PID feedback parameters are not optimized, a variable force is exerted on the surface by a probe during a scan.

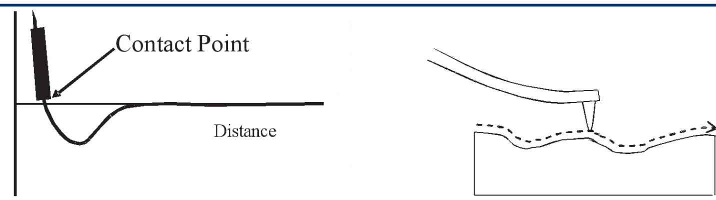


FIGURE 4-5 Left: Potential diagram showing the region of the probe while scanning in contact mode. Right: In contact mode the probe glides over the surface.

The forces applied to the surface by the probe in contact mode are given by Hooks law:

Equation 4-1:	$F = -k^{*}D$	F= force k= force constant D= Deflection distance

The force constant may be calculated if the dimensions and material of the cantilever are known. Most commercially available cantilevers for the AFM are supplied with the approximate values for the force constant. However, there is typically a very large error in the force constant because of the uncertainties in the thickness of the cantilever. If it is important to know the exact force between the probe and surface, it is recommended to use the Sader[5] method. In this method the length and width of the cantilever are measured with an optical microscope. The Q of the cantilever is measured and then the force constant can be calculated.

Contact mode is typically used for scanning hard samples and when a resolution of greater than 50 nanometers is required. The cantilevers used for contact mode may be constructed from silicon or silicon nitride. Resonant frequencies of contact mode cantilevers are typically around 50 KHz and the force constants are below 1 N/m. Figure 4-6 illustrates a few of the many examples of contact mode AFM images:

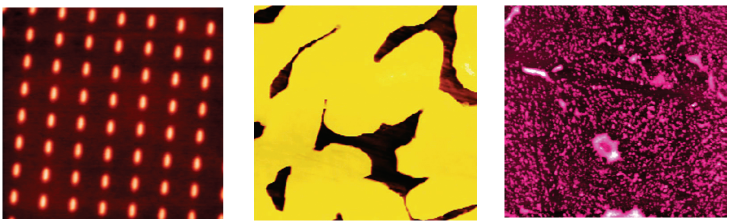


FIGURE 4-6 Contact mode images: Left: Bits on a compact disk. Center: Image of a metal surface. Right: Nano-particles on a surface.

4.1.2 Vibrating Modes

In order to make more sensitive measurements requiring better signal/noise ratios in scientific instruments, it is common to modulate the signal being measured and use phase or amplitude detection circuits. Use of modulated techniques shifts the measurement to a higher frequency regime where there is less than 1/f noise. Such techniques were developed for the AFM soon after it was invented.

In order to make the S/N ratio higher, and thus be able to measure lower forces with the AFM, the probe is vibrated as it is scanned across a surface. As shown in Figure 4-7, the probe is vibrated in and out of surface potential. The modulated signal can then be processed with a phase or amplitude demodulator.

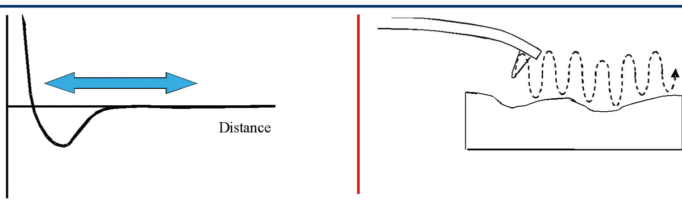


FIGURE 4-7 Left: Potential diagram showing the motion of the probe in vibrating mode. Right: The probe vibrates as it scans across a surface.

As illustrated in Figure 4-8, the cantilever can be excited with a piezoelectric ceramic. The cantilevers have natural resonant frequency ω_o given by:

Equation 4-2:	$\omega_o = c\sqrt{k}$	c = proportionality constant k= force constant

At the resonance frequency, there is a 90 degree phase shift. When the probe tip interacts with a surface, the resonance frequency shifts to a lower value, and there is a corresponding change in the phase. When scanning in the vibrating modes, a constant relationship is maintained by the feedback electronics, which keeps either the phase shift or amplitude constant at a given frequency, while scanning.

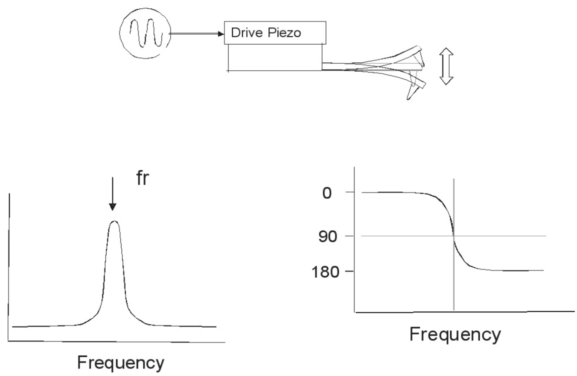


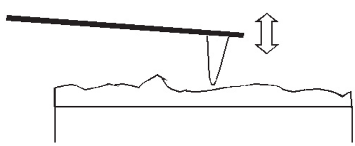
FIGURE 4-8 Top: A drive piezo causes the cantilever to resonate. Left: At resonance (fr) there is a large increase in the vibration amplitude of the cantilever. Right: At resonance there is a 90 degree phase shift in the cantilevers motion.

As mentioned in Section 4.0, there is a "contamination" layer on surfaces in ambient air with a thickness between 1 and 50 nanometers. The probe surface interaction forces are governed by the capillary forces between the probe and the contamination layer. The probe may be vibrated in three separated regimes as it is scanned across the surface, see Figure 4-9.

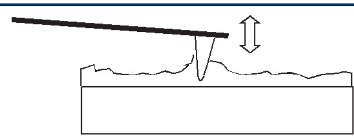
In the first regime, the probe is vibrated across the surface of the contamination layer. The vibration amplitude must be very small and a very stiff probe must be used. The images of the surface contamination layer are typically very "cloudy" and appear to have low resolution. This is because the contamination fills in the nanostructures at the surface.

In the second regime the probe is scanned inside the contamination layer[6]. This technique, named “near contact”, requires great care to achieve. The cantilever must be stiff so that the tip does not jump to the surface from the capillary forces caused by the contamination layer. Then very small vibration amplitudes must be used. Often very high resolution images are measured in this regime.

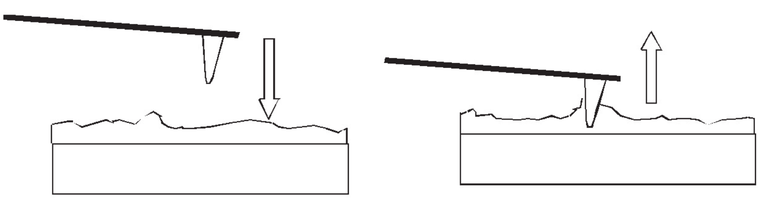
In the third regime the probe is vibrated in and out of the contamination layer. This mode is given several names such as intermittent contact[7] or tapping. In this mode the energy in the vibrating cantilever is much greater than the capillary forces and the probe moves readily in and out of the contamination layer. This mode is the easiest to implement but often results in broken probes because the tip is crashing into the surface upon each oscillation.



Case 1: The probe tip vibrates at the surface of the contamination layer.



Case 2: The probe tip vibrates inside the contamination layer.



Case 3: The probe tip vibrates from a position above the contamination layer to the surface and then back out of the contamination layer.

FIGURE 4-9 . Three scenarios for a vibrating probe in relationship to a surface coated with contamination.

One of the unintended benefits of vibrating the probe is that lateral forces that may be acting on the probe during scanning are released. That is, on each oscillation, when the probe is away from the surface, there are no horizontal forces on the probe. This concept was first identified for stylus profilers where it was proposed that vibrating the probe would reduce lateral forces.

Vibrating methods are used when the highest resolution is required or if very soft samples are being scanned. The probes used for vibrating mode are often less than 10 nm in diameter. The integrity of the probe during scanning at these high resolutions can be monitored with a "tip check" sample (see Figure 4-10).

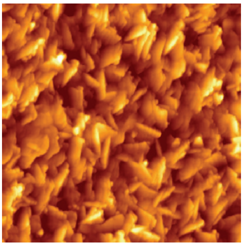


FIGURE 4-10 2X2 micron AFM image of a tip checker sample.

Most of the extremely high resolution images measured with an AFM in ambient air are made with vibrating mode. Figure 4-11 illustrates images measured with vibrating mode:

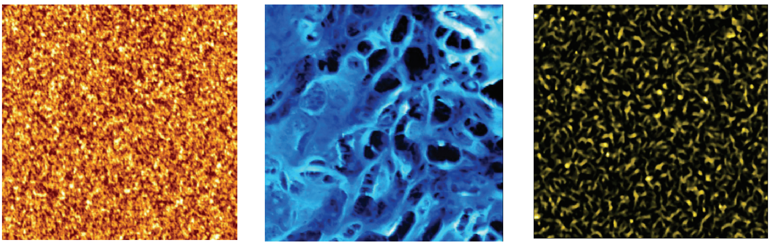


FIGURE 4-11 Vibrating mode AFM images. Left: Silicon wafer. Center: Cancer cells. Right: Proteins.

4.2 Field Modes

Field modes are used to measure the electrostatic or magnetic fields above a surface. Typically, the vibrating mode discussed in Section 4.1.2 is used to measure the surface fields. That is, the probe is scanned above the surface such that the probe interacts with the electrostatic or magnetic fields that emanate from the surface.

The shift in resonant frequency of a cantilever that is vibrating in a field is given by[8]:

Equation 4-3: $$\omega_o - \omega_o' \approx \omega_o \, {}^{f'}\!/_{2k}$$

Equation 4-4: $$f' = \frac{dF}{dz}$$

k	Force constant
ω_o	Resonance frequency
ω_o'	New resonance frequency
F	Force on probe

To get the change in vibration amplitude due to a field at the surface, the first derivative of the force with respect to the distance from the surface must be calculated.

Images of surface fields are typically measured by making two scans of the surface. In the first scan, the topography of the surface is established. In the second scan the probe is scanned at a fixed distance from the surface (see Figure 4-12).

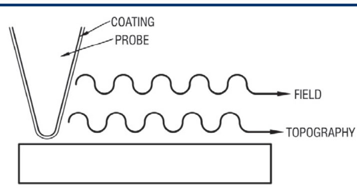


FIGURE 4-12 Topography is measured when the probe is vibrated near the surface, When the probe is vibrated at a fixed distance from the surface, surface fields are measured.

There are several more sophisticated techniques for measuring surface fields such as oscillating the force setpoint, or actually moving the probe up and down as it is scanned across the surface, illustrated in Figure 4-13.

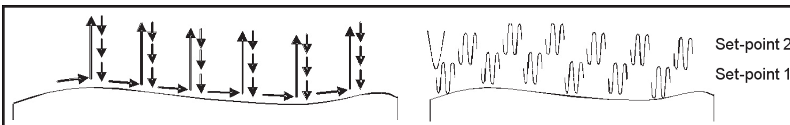


FIGURE 4-13 Methods for separating the topography information from the field information in the field modes. Left: Hosaka Method. Right: Set-point Method.

The field modes require a tip that is coated with either an electrically conductive or magnetic material. Because the probes are coated, the resolution of the field modes are typically much less than the resolution of the topography modes. The decrease in resolution is correlated to the thickness of the coating. The success of making field measurements ultimately depends on getting a high quality coating on a probe. The coatings can be damaged while scanning if the probe contacts the surface.

4.2.1 Electric Force Microscopy

The equation for electrostatic forces between a probe and a surface having different potentials is given by[9]:

Equation 4-5: $$F_{electrostatic} = -\frac{1}{2}\left(\Delta V\right)^2 \frac{dC}{dz}$$

ΔV Probe/Sample voltage difference
C Capacitance

It can be determined from Equation 4-5 that the change in resonant frequency is proportional to the derivative of the force. Thus, provided that the potential between the probe and surface is not zero, the change in the first derivative of the force is proportional to the changes in capacitance as a function of the second derivative of Z spacing.

Figure 4-14 illustrates the effect of an electric field on a vibrating cantilever as it is scanned across a region of electric charge. The magnitude and direction of the amplitude changes, or resonance frequency shift, depends on the polarity of charges on the sample and the polarity of charges on the probe.

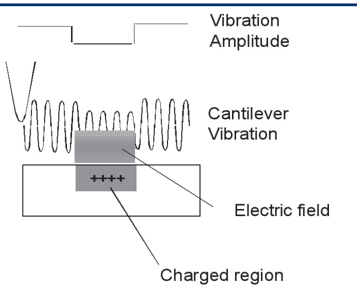


FIGURE 4-14 The probe vibration amplitude at a set frequency changes as the probe moves through the electric field.

4.2.2 Magnetic Force Microscopy

In a Magnetic Force Microscope (MFM), a probe coated with a magnetic film is vibrated slightly above a surface while scanning as illustrated in Figure 4-15.

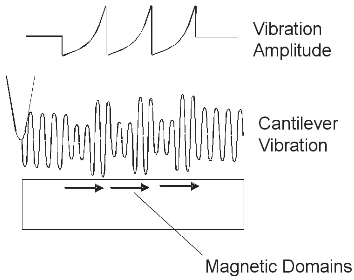


FIGURE 4-15 As the probe passes though the fields generated by the magnetic domains, its vibration amplitude changes.

In a hypothetical ideal case of a single monopole on surface and a single monopole on a probe, the equation for the derivative of the force is given by[10]:

Equation 4-6: $$f' = \frac{6\mu_o m_p m_t}{\pi (s + z_o)^5}$$

Equation 4-7: $$z_o = \frac{R}{2} + \frac{d_m}{2} + \delta$$

s	tip surface distance
—	nonmagnetic thickness
R	tip diameter
mp	magnetic moment particle
mt	magnetic moment tip
dm	mean magnetic diameter

Thus, the general trend is that the magnetic force signal changes with the inverse of distance between the probe and the surface to the 5th power. Figure 4-16 shows MFM images of a magnetic tape.

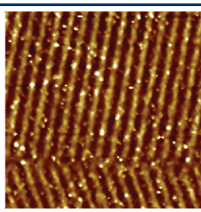


FIGURE 4-16 MFM image of magnetic tape.

As with EFM, the resolution achievable with MFM depends on the thickness and quality of the coating at the apex of the probe. This is often very difficult to control so the results from MFM are fairly inconsistent. Also, when trying to measure the magnetic domains on a soft magnetic material, the domains on the probe can cause a change in the domain structure on the surface.

4.3 Material Sensing Modes

An AFM probe is sensitive to changes in the physical properties at a sample's surface. The physical properties include the chemical composition, the hardness, and the adhesion. Images of surface physical properties measured with the AFM are qualitative and show changes in the physical property. The difficulties in getting quantitative values for physical properties are derived from the inability to absolutely control the probe geometry and chemical properties of an AFM probe.

4.3.1 Lateral Force / Frictional Force

As illustrated in Figure 4-17, the cantilever in an AFM can twist or rotate as it is scanned across a surface. The amount of motion is in some way related to the differences in the chemical/physical properties of a surface. The chemical/physical property could be the nano-roughness or it could be differences in chemical composition.

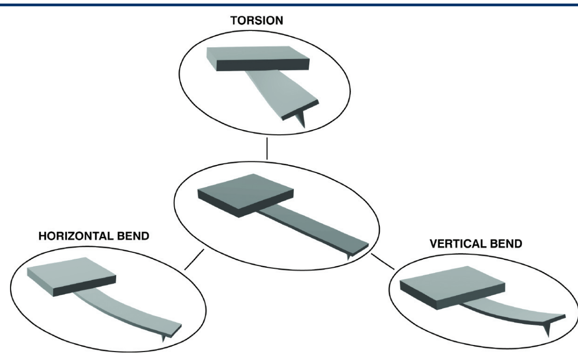


FIGURE 4-17 Lateral forces on the tip of an AFM cantilever can cause the cantilever to bend vertically (lower right), bend horizontally (lower left), or twist (upper).

Typically, the force constant coefficient for twisting in the lateral direction is much less than the force constant coefficient for bending. Thus, the cantilever tends to twist while scanning. Because the physical dimensions of the cantilever are not well characterized in commercially available cantilevers, the force constant coefficients must be calculated for each cantilever. Calculations are derived from the physical dimensions and the cantilever's material.

The twisting of the cantilever is measured in the light lever AFM by monitoring the left to right motion of the deflected laser light with a photodetector. Typically, the photodetector has four quadrants. The vertical force is measured by monitoring the top and bottom two quadrants, and the lateral forces are measured by monitoring the left and right two quadrants. Figure 4-18 illustrates the sensing system used for measuring lateral motions of the cantilever. Both a topography and frictional image may be measured simultaneously.

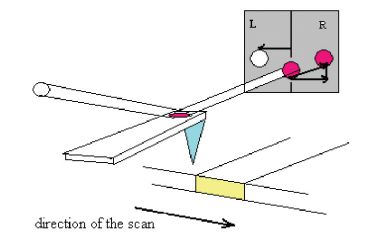


FIGURE 4-18 In LFM the probe torsional movement is monitored with a four section photodetector having a left (L) and right (R) section.

Figure 4-19 is an example of lateral force images on a very flat piece of gold. The topography image of this sample shows no features, however the LFM image shows a pattern drawn on the surface.

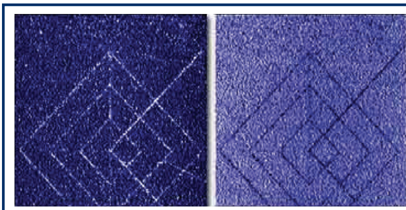


FIGURE 4-19 LFM image of a flat sample patterned using AFM lithography. At the left is the LFM signal scanning in one direction, at the right is the LFM signal scanning in the other direction.

4.3.2. Vibrating Phase

Physical mechanical properties such as the surface hardness or stiffness of materials can be directly measured with the vibrating phase technique. When scanning a surface in vibrating mode, the feedback either keeps the relative phase or vibration amplitude of the cantilever constant (see Section 4.1.2). Thus while scanning, if the probe traverses a surface area having different mechanical hardness (stiffness), and the vibration amplitude is held constant, there will be a change in the phase signal. Figure 4-20 illustrates the operating principle of vibrating phase mode.

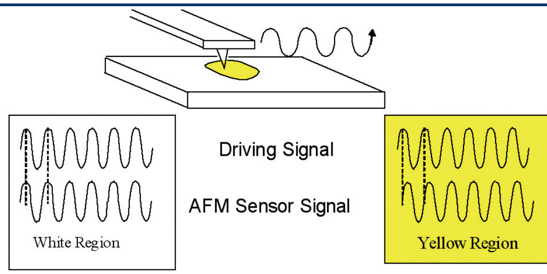


FIGURE 4-20 Vibrating Phase Mode, as the probe goes over the yellow region the phase shift between the "drive signal" and the "AFM sensor signal" changes.

Vibrating phase images are measured simultaneously with topography images. Although vibrating phase mode gives excellent contrast on many samples, extracting quantitative data is very difficult. This is because the exact nature of the probe sample interaction is not well known. Many of the common applications for vibrating phase mode are for studying polymers and composites (see Figure 4-21).

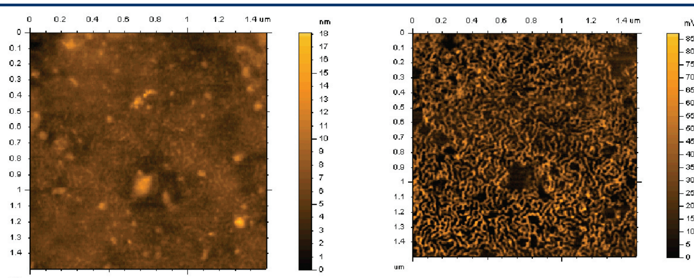


FIGURE 4-21 Left: Topography. Right: Vibrating phase mode image of the SBS triblock co-polymer.

4.4 Electrical Modes

A conductive AFM probe may be used for measuring the electrical properties of a surface or structures located on the surface. The probe itself can be conductive or it can be made to be conductive by coating it with a metal layer. It should be pointed out that the electrical modes include EFM (discussed in Section 4.2.1).

Although the electrical modes are typically relatively simple to implement, getting reliable data is problematic. There are three problems:

a) The measurements are typically made on materials that are coated with contamination, and getting a reliable electrical contact is difficult.

b) If the probe is coated with a metal, the metal coating can be removed when the probe contacts the surface.

c) An oxide layer can build up on the tip or surface making the measurement unreliable.

Figure 4-22 illustrates a probe in the proximity of a sample's surface and the layers of oxide and contamination that cause uncertainty in AFM electrical measurements. The oxide layers and contamination make it difficult to make an ohmic contact between the probe and surface.

One approach to solving issues b and c is to coat the probe with a conductive diamond coating. The diamond coating is very hard and does not oxidize; however, the coating increases the diameter of the probe significantly. Even when the probe is coated with an inert layer such as diamond, there are still potential problems for contamination.

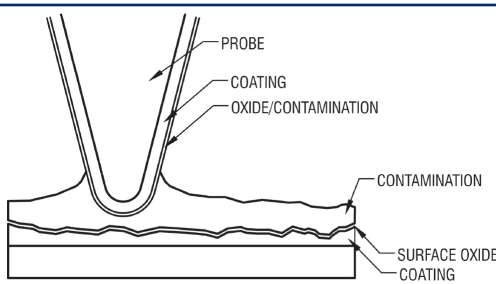


FIGURE 4-22 Electrical measurements with the AFM require the probe to make electrical contact with the coating.

4.4.1 Parametric Testing (I/V and C/V)

An electrically conductive probe may be used directly for parametric testing (see Figure 4-23). The parametric tester is attached directly between the conductive AFM probe and the sample. When measuring I/V curves, a current-limiting resistor is often required to prevent the probe tip from being destroyed by too much current.

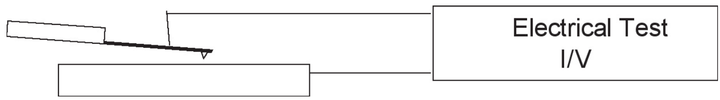


FIGURE 4-23 Parametric testing is possible by electrically connecting the parametric tester to a conductive AFM probe.

Although this technique initially seems promising for measuring electrical properties of nanostructures such as nanotubes or quantum dots, there are a few drawbacks. In addition to the problems listed in Section 4.4.0, there is another major drawback. The absolute positioning accuracy of an AFM scanner is limited by thermal drift in the stage. When scanning, it is possible to measure structures with a few nanometer dimensions, although it is very difficult to absolutely position the probe over a feature that has only a few nm in diameter.

4.4.2 SHARK

The spatial map of the electrical conductivity of a surface is measured with SHARK. The apparatus for making a SHARK map is illustrated in Figure 4-24. A bias is placed between a conductive sample and probe. Then the sample's surface is scanned. Monitoring the current flow between the probe and the surface yields a conductivity map of the surface. A topogram of the surface's 3-d structure is measured at the same time as the conductivity map.

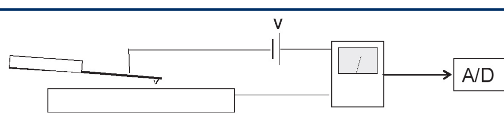


FIGURE 4-24 Electrical conductivity maps are measured by monitoring the current flow between the probe and the surface. The image is displayed with the A/D converter.

Often, a current limiting resistor is necessary to limit the current flow.

Excessive current can heat and destroy the apex of the probe. The value of the current limiting resistor depends on the conductivity of the sample being analyzed. The SHARK electronic unit provides for selection of a current limiting resistor as well as the bias between the probe and sample.

Figure 4-25 is an example of SHARK made on a carbon nanotube that is held perpendicular to a sample's surface.

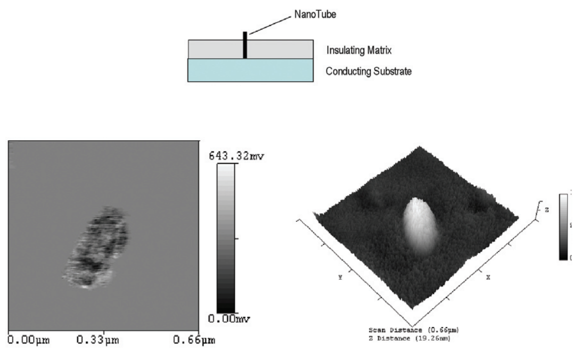


FIGURE 4-25 Example of SHARK. A carbon nanotube is placed vertically on a conducting substrate and held in place with an insulating matrix. The probe is then scanned over the surface and an electrical conductivity map (left) and a topography map (right) are generated.

4.4.3 Ferroelectric / Piezoelectric Testing

Ferroelectric/piezoelectric materials have unique electrical and physical properties that can be measured with an AFM probe. The advantage of the AFM probe for ferroelectric measurements is that the probe has high spatial resolution and localized measurements are possible. There are two primary types of measurements made on ferro/piezo films: electrical properties and spatial displacement.

The electrical properties are measured with a parametric tester and apparatus similar to that described in Section 4.4.1. The probe is placed in contact with the ferro/piezo film and the electrical measurement is made.

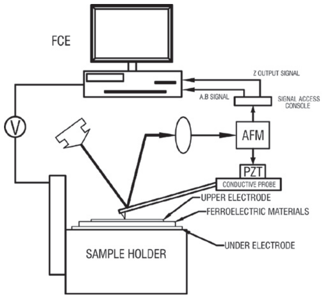


FIGURE 4-26 Apparatus used for AFM FE-RAM testing.

The ferroelectric or piezoelectric response of a film is measured by monitoring the motion of the cantilever when a bias is applied to the film with the apparatus illustrated in Figure 4-26. A butterfly curve corresponding to the electrical excitation of the ferro/piezo film (Figure 4-27) is measured. The spatial resolution of the measurement is proportional to the probe diameter.

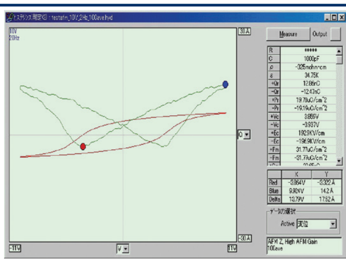


FIGURE 4-27 Butterfly curve of ferroelectric sample measured with an AFM (green).

4.4.4 Kelvin Probe (SKPM)

The force between an electrically conductive probe and a sample is given by Equation 4-5. The force will be zero if the potential of the probe (Vp) and the sample (Vs) are equal. In the Kelvin probe mode, the probe is scanned across a surface, typically in vibrating mode, and a feedback loop is used to keep the voltage between the probe and surface equal.

The feedback loop used to control the potential between the probe and surface is zero and is illustrated in Figure 4-28. The mechanical vibration mode is w1 and the probe potential is vibrated at another frequency w2. The signal at the photodetector is modulated at both frequencies; w1 is generated from mechanics, and w2 is generated from varying electric potential. When the force between the probe and surface is minimized (or zero) there will be no modulated signal at w2. A feedback electronic circuit is used to maintain the potential of the surface such that Vp = Vs.

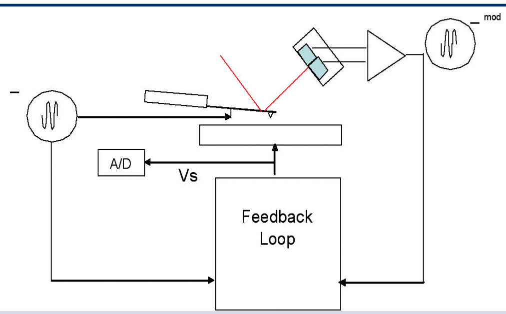


FIGURE 4-28 Apparatus used for measuring surface potential with an AFM. The mechanical vibration frequency w1 is not presented in this figure.

Scanning Kelvin Probe Microscopy is a quantitative technique and measures the absolute potential of surface. In contrast, EFM (see Section 4.2.1) measures electric fields and is qualitative. Additionally, because SKPM uses the vibrating mode to track topography, there is less force placed on the surface and the integrity of the probe stays intact. However, SKPM is a more costly technique to implement because it requires a lock-in amplifier and PID controller.

Figure 4-29 illustrates the use of a KPM for imaging the surface of a phase recorded optical medium. The topography image does not show the bits on the surface, however, the bits are very apparent in the potential image. Both the topography and potential image are measured simultaneously.

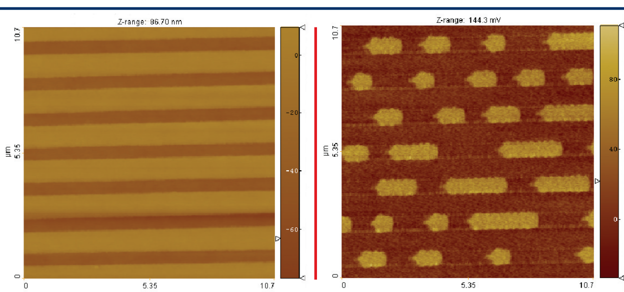


FIGURE 4-29 Topography (left) and Potential (right) image of a CD-R/W sample.

4.4.5 Scanning Capacitance

In the scanning capacitance microscope, the capacitance between the probe tip and surface are monitored. SCM is implemented by connecting a capacitance sensor between the probe and surface while scanning (see Figure 4-30). Alternatively, the Kelvin probe technique can be adopted to measure capacitance profiles. The Kelvin probe technique is adapted by monitoring the w2 signal with a lock-in amplifier.

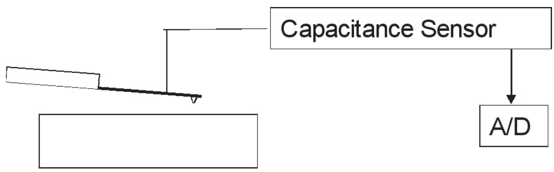


FIGURE 4-30 A capacitance sensor measures the capacitance between the probe and sample. The image is output to an A/D converter.

Although SCM has great promise for applications, especially in the semiconductor industry, there are a few problems. First, the reliability of the data measured is not good because of surface contamination and uncertain probe geometries. Second, data modeling is difficult and getting the resolution required for applications such as dopant profiling at the nanometer scale are not possible.

4.5 Lithography

The probe in the AFM can be used for making nanoscale changes to a surface's physical structure or nanoscale chemical composition. There are several methods for making changes in a surface including: voltage induced, mechanically induced, and chemical deposition (see Sections

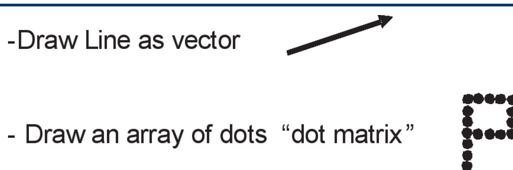


FIGURE 4-31 Two methods for writing on a surface with an AFM probe.

4.5.1, 4.5.2, and 4.5.3). Regardless of the method of initiating the surface change there are two basic types of lithography possible with the AFM; they are a vector and dot mode (see Figure 4-31).

The patterns that are created on a surface with an AFM in lithography mode are typically generated from an input file, typically a .bmp or .jpg file. After inputting the pattern, the software generates the motions required for the probe to create the pattern on a surface. Associated with the pattern is the method for writing, the writing speed, and the mode of writing. Figure 4-32 illustrates a typical software input window. In general, the resolution of AFM lithography techniques is proportional to the diameter of the probe used for generating the pattern.

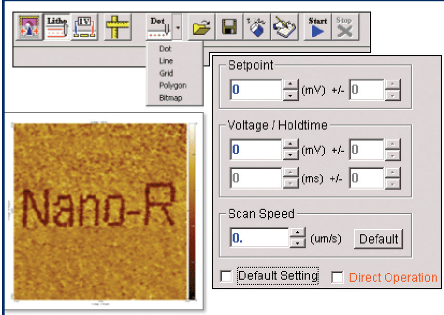


FIGURE 4-32 AFM lithography software inputs the pattern to draw and allows selection of the type of patterning.

The AFM affords one of the most economical platforms for creating nanoscale features on a surface. The versatility of the tool for lithography is substantial. However, there is one major drawback; the scan rates of an AFM are slow. Generating a pattern can be very time consuming with an AFM.

4.5.1 Voltage

In 1989 it was demonstrated that the localized current from a small probe, as in a scanning tunneling microscope, can cause changes in a material's surface[11]. In an AFM, current flowing from the apex of the probe to the sample can cause chemical reactions at the surface.

The most common example of AFM lithography using a potential between a probe and surface is anodic oxidation. Typically a potential is placed between a probe and a silicon wafer (see Figure 4-33). An electrochemical reaction occurs at the surface of the silicon. In this example, the width of the line depends on the number times the line is traced.

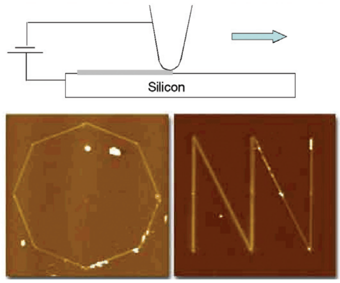


FIGURE 4-33 Example of voltage generated lithography pattern in an AFM. At the bottom is an example of anodic oxidation with an AFM. The lines are 50 nm wide.

4.5.2 Scratching

The force of the probe on the surface can be increased while scanning with an AFM. If the sample's surface is softer than the probe, the probe will scratch the surface. Figure 4-34 shows an image of a pattern drawn in the surface of photo resist with an AFM.

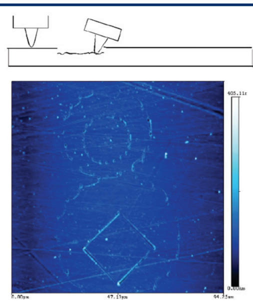


FIGURE 4-34 An AFM probe can scratch a surface. The image at the bottom shows a pattern with nm features created by scratching.

4.5.3 Chemical Deposition

Chemicals may be deposited directly from an AFM probe onto a surface[12], see Figure 4-35. The chemicals may flow by diffusion from the surface of a probe. Alternatively, the chemicals may be deposited on a surface from a hole fabricated in the probe. It has already been demonstrated that several types of chemicals may be deposited on a surface. Examples include thiols on gold, proteins, and metals.

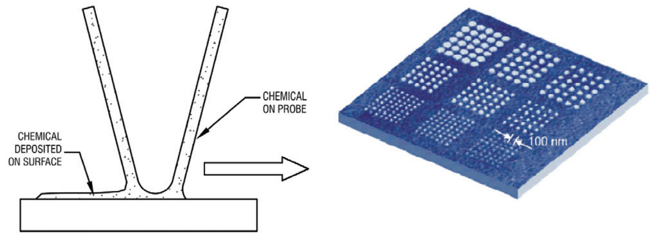


FIGURE 4-35 Chemicals may be deposited from an AFM probe onto a sample's surface. At the right is an example DPNTM for creating an array of nanodots.

4.6 Mechanical Measurements

The probe in an AFM may interact with the hard forces at a surface. Such interactions allow making nano-mechanical measurements on a surface. The primary method for making mechanical measurements is the force/distance curve. When the probe is pressed firmly into a surface, it may cause a nano-indentation of the surface.

4.6.1 Force / Distance Curves

A force/distance (F/D) curve is a measure of the forces on the probe as a function of the distance of the probe from a sample's surface. Typically a F/D measurement is initiated with the probe in free space above the surface. Then the probe is lowered towards the surface until it interacts with the hard force in the repulsive regime. Then the probe is pulled away from the surface until it is in free space again. A force distance curve and the corresponding positions of the probe are illustrated in Figure 4-36.

As the probe begins to interact with the surface, it is pulled into the surface by capillary forces. This process is called jump to contact and often this step breaks the tip off the probe. The position of the jump to contact is dependent on the thickness of the contamination layer on the surface. When retracting as the probe is pulled from the surface, the adhesion causes the probe to "stick" to the surface, and then disengage from the surface.

Errors can occur in the F/D curve if a position sensor is not used to measure the motion in the Z axis. The error occurs because the Z ceramic generating the motion is not linear and has hysteresis. Such an error appears as a displacement in the F/D curve when the probe is in the repulsive region of the curve.

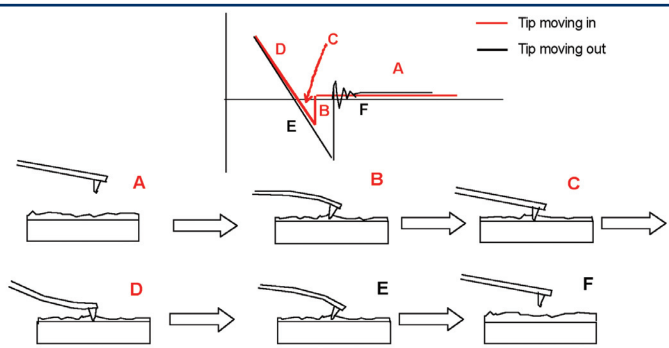


FIGURE 4-36 The force distance curve is measured by moving the probe at the end of a cantilever toward a surface (red and black traces at the top). The letters A-F indicate the position of the probe as it moves toward and away from the surface.

4.6.2 Nano Indenting

A nano-indent in a surface can be generated by physically forcing the probe into a surface, causing surface material to be dislodged. These "loading curves" can be created only if the spring constant of the AFM cantilever is stiff enough, and the probe material hard enough. Figure 4-37 shows a nano-indent created by a silicon probe in the surface of PMMA.

FIGURE 4-37
Nano-indentation made with an AFM probe in PMMA surface.

There are two problems that make it difficult to get quantitative data when nano-indenting with an AFM. The first is that the probe geometry is not well known or at best poorly defined. Secondly, as seen in Figure 4-38, the probe sample angle is constantly changing as the nano-indent process is being made.

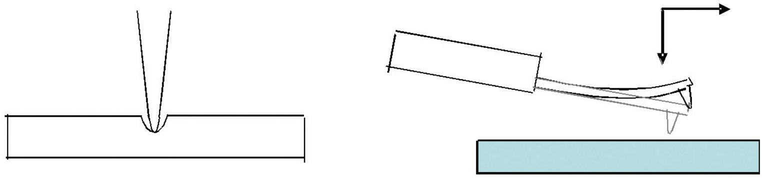


FIGURE 4-38 Left: Quantitative nano-indentation measurements require that the probe approach in a purely vertical motion. Right: Because the probe is at the end of a cantilever in an AFM, the probe approaches the surface at an angle.

4.6.3 Frictional Measurements

The frictional coefficient of a probe moving over a surface is defined as the loading force divided by the horizontal force:

$$u = F/N \quad \text{(Equation 4-8)}$$

Because an AFM can measure the vertical and horizontal force (see Section 4.3.1), it is possible to measure frictional coefficients at the nanoscale. A frictional loop curve is first measured (see Figure 4-39); then the coefficient of friction which is proportional to W may be calculated[13]. Calculation of the frictional coefficient from a frictional loop requires detailed knowledge of the parameters associated with the geometry of the AFM scanner from which the measurements are derived.

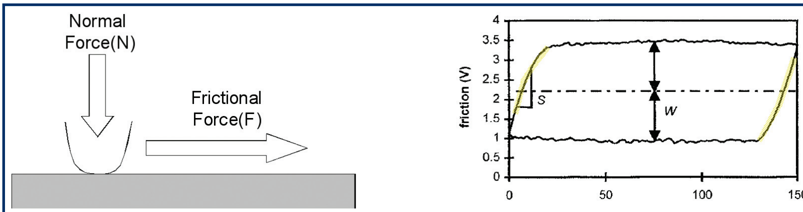


FIGURE 4-39 Left: The frictional coefficient is defined as the frictional force divided by the normal force. Right: A frictional loop is measured by monitoring the lateral signal as it goes from left to right and right to left.

4.7 Thermal Measurements

By placing a small temperature sensing device at the end of an AFM probe it is possible to make a number of thermal measurements of a surface. The two primary types of probes used in an AFM for making thermal measurements are the thermocouple and the resistive probe, shown in Figure 4-40.

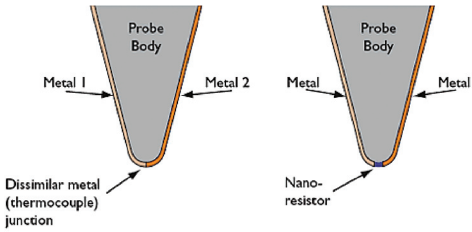


FIGURE 4-40
Left: Probe made with a thermocouple.
Right: Probe made with a small resistor.

The types of measurements that are possible with an AFM having a thermal probe are:

a) Surface Temperature – With both the thermocouple and resistive probe it is possible to measure surface temperature maps.

b) Surface Thermal Conductivity Maps – With the resistive probe it is possible to measure the cooling of the probe as it traverses a sample. The cooling is proportional to thermal conductivity of the sample.

c) Thermal Analysis Maps – A temperature ramp may be applied to the resistive probe. As the probe heats, its vertical motion may be measured as the surface melts, and the transition temperatures may be established.

4.8 Other Modes

There are a number of modes that are possible with an AFM that were not covered in the previous sections. The modes and a brief description follow:

4.8.1 Electrochemistry

An electrochemical cell may be added to an AFM so that electrochemistry experiments can be made in situ. The AFM can then be used to study electrochemical changes in surface properties without the surface being exposed to air. With the addition of a galvanonstat, the surface topography can be investigated as a function of the surface potential relative to a reference cell.

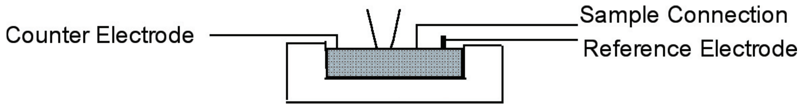


FIGURE 4-41 The AFM probe may be used to study samples in an electrochemical cell.

4.8.2 Scanning Tunneling Microscope

The scanning tunneling microscope was invented 5 years before the atomic force microscope. However, the applications for STM are for the most part limited to studies of atomic structure in an ultra high vacuum chamber.

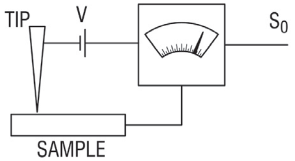


FIGURE 4-42 An AFM may be adapted to make STM measurements. In an STM the current between the probe and sample is used to control the distance between the probe and sample.

4.8.3 Pulsed Force Mode

With pulsed force mode surface topography, adhesion and stiffness are measured simultaneously. PFM is implemented by placing a sinusoidal voltage on the Z piezo and monitoring the light lever output at key points.

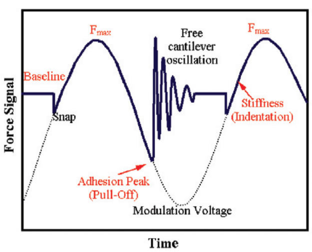


FIGURE 4-43 In pulsed force mode the probe is moved in the Z axis in a sinusoidal motion.

4.8.4 Nano-Manipulation

An AFM probe may be used to move nanoscale objects on a surface. The motion may be performed in an open loop format with special software, or in a closed loop format with a haptic interface.

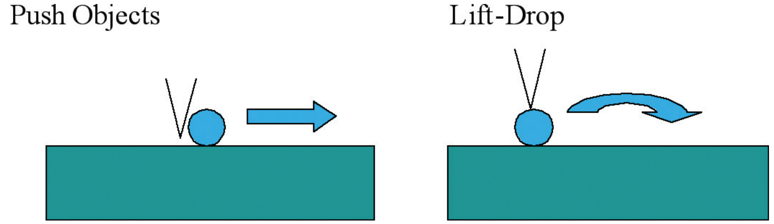


FIGURE 4-44 An AFM probe may be used to move objects on a surface. The objects may be pushed or they maybe lifted or dropped onto the surface.

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CHAPTER 5

Process, Display, Analyze Software

AFM images are stored in a computer as a three dimensional array of numbers. The array of numbers can be processed, displayed, analyzed and then reported by specialized image processing software. AFM image processing software is typically just a little different than image display software that is used for traditional microscopes, such as optical and scanning electron microscopes. The major difference is that the AFM images have three dimensional topography content and the traditional microscopes do not; i.e., traditional microscopes do not have height information.

Definitions for the various functions of AFM image processing software are:

Process: Changes the data in the image and includes functions like filtering and background subtraction.

Display: Changes the view of the data on the computer screen and includes the type of colors used and the perspective of the display.

Analysis: Achieves the abstraction of quantitative information such as line roughness.

Report: Images and analysis dialog boxes may be exported to a clipboard or to Microsoft™ Office products with the report functions.

The following sections describe the primary functions in each of these steps.

5.1 Process

5.1.1 Leveling

AFM images always have some background slope or curvature that must be removed from the image. Sources of the background can be an offset angle between the probe and surface, or curvature introduced into the image from the xyz scanner. There are several algorithms that are used for "leveling" the images. The primary methods are:

Line: Line by line leveling is the most common method for leveling AFM images. In this method each horizontal, or vertical, line in an image is fit to a polynomial equation, and then the polynomial shape is subtracted from image line. Then, the average height of each line is set equal to the previous line (see Figure 5-1).

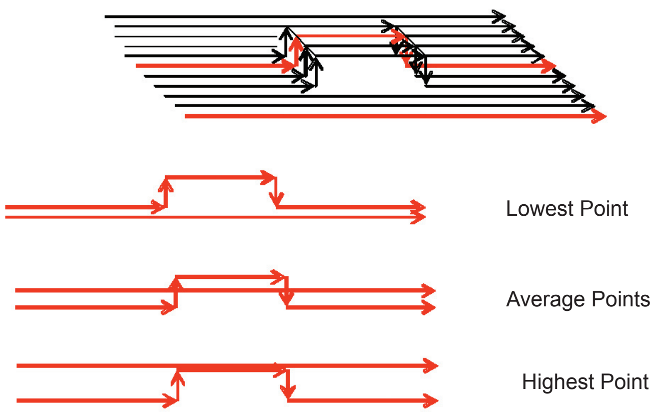


FIGURE 5-1 Line by line leveling is the simplest method for removing unwanted background bow and tilt from AFM images. A line is "fit" to each of the scan lines, and then the "fit" line is subtracted from the scan line.

Three point: In the three point method, the AFM operator identifies 3 points on an image. The three points define a plane which is then subtracted from an image. Three point leveling is ideal for samples that have terraces where the background bow associated with the scanner is much less than the height of the terraces.

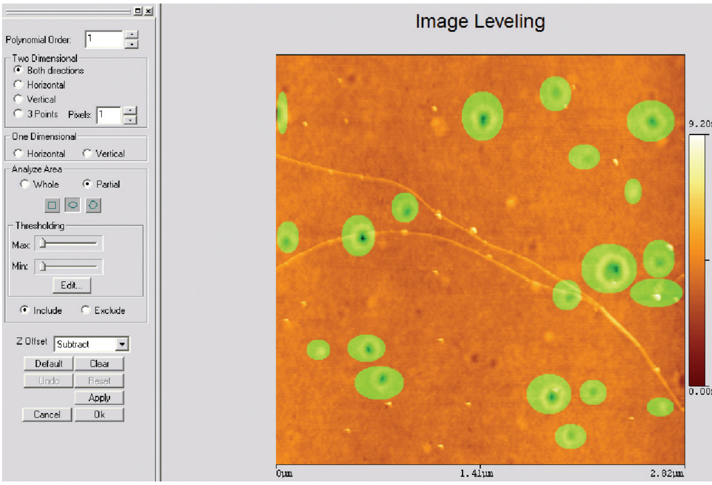


FIGURE 5-2 Areas of an image may be selected and then the background removal, or leveling, does not use these areas in background subtraction calculations.

Inclusion/Exclusion: Often AFM images have a few isolated features on a very flat surface. If line leveling is used, the features cause streak marks in the image (see Figure 5-2). This "artifact" can be overcome by using the inclusion/exclusion feature in the image analysis software. The operator identifies the features to be included or excluded in the line by line leveling. When the line leveling is done, the "marked" features are not used for the calculation of the polynomial line that is subtracted from the image.

5.1.2 Histogram Adjust

A histogram of an image is created by making a plot of the number of pixels in the image versus the color of the pixels (see Figure 5-3). The histogram adjust function allows the distribution of a broad range of colors across the entire color scale. This option can be used for showing surface features that are not visible when the color scale takes the entire Z range of the image.

Often after an image is histogram adjusted, features can be visualized and measured in an image that could not be observed in the original image.

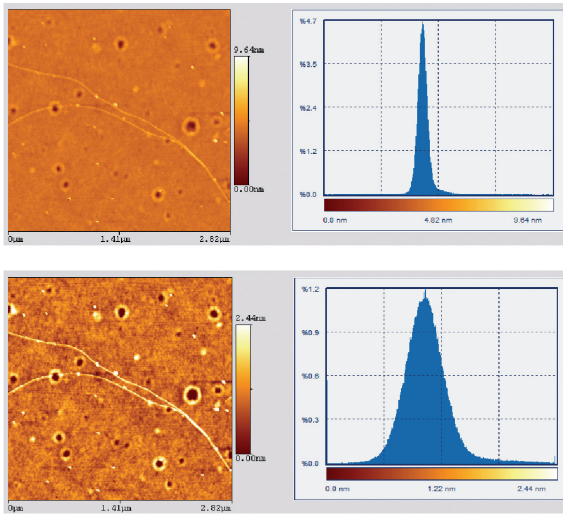


FIGURE 5-3 With the histogram adjust function the color scale distribution is spread across more of the available color scale. In the top image, a very small amount of the available color scale is used for features in the image. After the "histogram adjust", more of the color scale is used for features in an image. Often, by adjusting the histogram, it is possible to see more structure in an AFM image.

5.1.3 Filtering

Often there is unwanted high and low frequency noise that appears in AFM images. This noise can be removed by filtering. The two types of filtering most commonly used on AFM images are Matrix Filtering and Fourier Filtering.

Matrix Filtering averages 2, 3, 4, 5, 6 adjacent points together in an image. The averaging may be weighted such that the data points directly next to the pixel being calculated has more weight than the others. Matrix filters can be use to sharpen or to blur an image.

Equation 5-1:	$P2 = (P1 + P2 + P3)/3$	P1, P2, P3 are adjacent points in an image

Equation 5-1 would be the calculation for a single data point with a weighting value of 1 of each of the surrounding data points.

Fourier Filtering takes an image and calculates its frequency components, called the FFT image. Then unwanted frequency components are identified and removed from the FFT image. When the FFT image is back transformed, the resulting AFM image will not have the frequency components that were removed in the FFT image. FFT filtering is particularly effective on images with repetitive patterns.

5.1.4 Scale / Zoom / Rotation

An AFM image may have many features an operator does not want to show. These can be "removed" by zooming in on only the desired features. This is only possible if the original image has a high enough resolution that the resulting image has enough pixels. Associated with the zoom function, pixels may be mathematically added to the image with the scale function. As an example, if an image is 200 X 200 pixels and an area that is 50 X 50 pixels is used for the zoom, it is possible to increase the number of pixels in the final image from 50 X 50 to 200 X 200. Often, zooming will result in images that show a lot of pixilation.

Another process step that is sometimes required is to rotate the scan axis in an AFM image. This is necessary if it is required that features in an image line up with the scan axis. This is often required for the analysis of technical samples.

5.1.5 Error Correction

Unwanted errors sometimes occur in AFM images; for example there may be a short term unwanted vibration or a "glitch" in the image. These errors may be removed with special software for error correction. Line removal can be done by removing a single line and replacing it with the average of the two lines next to it. A "glitch" may be removed by replacing the glitched pixel with the average of the eight pixels around the unwanted pixel.

5.2 Display

A great amount of information is gleaned from an AFM image through visualization of the image. There are several ways of displaying images to make them more interesting to view. Sometimes features can be enhanced and seen more easily by changing the display parameters. Display functions do not change the AFM data or change the way the data is viewed.

5.2.1 2-D / 3-D

Images may be displayed by a computer as either 2-dimensional or 3-dimensional projections (see Figure 5-4). The two dimensional image shows the x and y axis and color is used to depict height. An AFM image displayed in the 2-D format looks much like an image from a traditional microscope such as an optical microscope.

An image displayed in a 3-D format gives a rendition of what the surface topography actually looks like. That is, data is displayed in the x y and z axis. Often the scale between the xy and z axes are not equal. The surface features are very small with relationship to the x and y dimensions; however, in the 3-d image they look large.

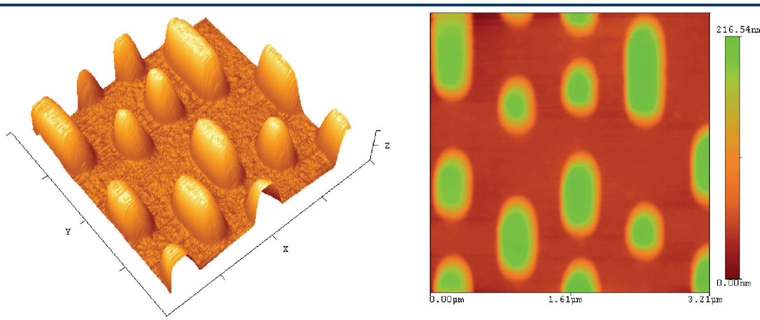


FIGURE 5-4 3-D presentation (Left) and 2-D presentation (Right) of an AFM image of DVD bits. The same data was used for both of these images.

5.2.2 Pallets

The color pallet used to display an AFM image can be selected to make the image seem more visually compelling. In some cases, selecting a specialized color pallet can help with visualizing certain aspects of an image. However, in general, the color pallet that is used for displaying an AFM image is very subjective.

5.2.3 Light Shading

Both 2-D and 3-D images can be viewed with light shading, as illustrated in Figure 5-5. By simulating a light shining on an image, the image looks photorealistic. The position and intensity of the light shining on the AFM image can be changed. Light shading often helps visualize the smallest, high frequency, structure on a surface.

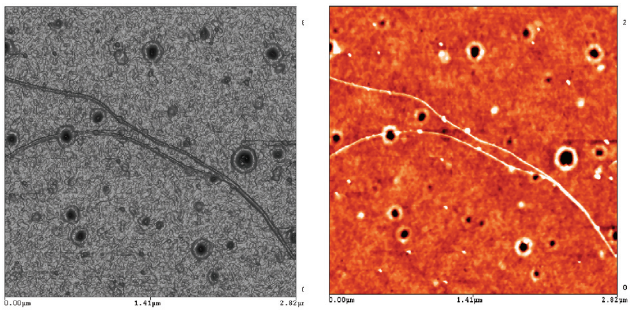


FIGURE 5-5 Light shaded (Left) and color scale (Right) image of carbon nanotubes. The light shaded image typically looks more photo realistic than the color scale image.

5.2.4 Contrast / Brightness

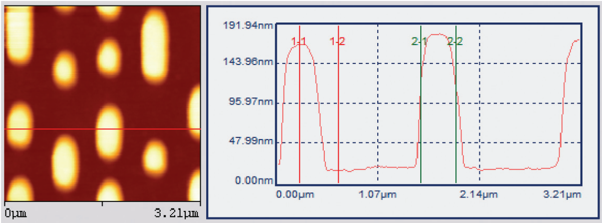
Contrast and brightness functions in an AFM image are the same as used in a video monitor.

5.3 Analysis

AFM images are visual representations of a three dimensional array of numbers, thus it is possible to make quantitative analysis of AFM images. Of course, the quantitative measurements derived from AFM images are only as good as the quality of the measurements. For example, if the images have errors due to poor scanner quality or dull probes, the analysis will reflect these problems.

5.3.1 Line Profile

A line profile is a two dimensional profile, or cross section extracted from an AFM image. The line profile may be taken horizontally, vertically or at an obtuse angle. Line profiles are the most common type of analysis made on AFM images. From a line profile, the distances between two points, as well as angle, may be calculated, (see Figure 5-6).



		Pair 1		Pair 2	
		X (µm)	Z (nm)	X (µm)	Z (nm)
Line1	1	0.228	169.469	1.514	98.600
	2	0.638	15.000	1.890	125.404
	distance	0.410	-154.470	0.376	26.804
	pt.angle	-20.65 °		4.08 °	

FIGURE 5-6 Line profile analysis of an AFM image of DVD bits. The height and with of the surface features are measured from the line profile.

5.3.2 Line / Area Roughness

The line and area roughness can be easily calculated from an AFM image. Standard equations, see below, are typically used for these calculations.

Equation 5-2: Surface Roughness
$$S_a = \frac{\sum_i^N \left| Z_i - \bar{Z} \right|}{N} \qquad \bar{Z} = \frac{\sum_i^n Z_i}{N}$$

Equation 5-3: Root Mean Square
$$S_q = \frac{\sum_i^N \left(Z_i - \bar{Z} \right)}{N}$$

Equation 5-4: Peak to Peak
$$S_r = Z_{max} - Z_{min}$$

Equation 5-5: Mean Value
$$S_m = \bar{Z} - Z_{min}$$

When comparing the line or area roughness it is important that the measurement is made on the appropriate length scale. Also, the values for line/area roughness depend on the length/size of the image, (see Figure 5-7).

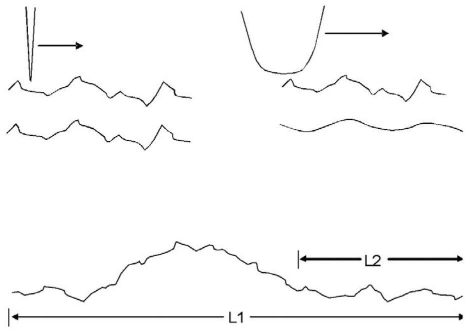


FIGURE 5-7
Errors in surface texture calculations can occur if (Top) the probe is not sharp enough to correctly track the surface topography and (Bottom) the scan range is not long enough to sample the extremes of the surface texture. In this case if the scan range is L2 a different value will be reached then if the scan range is L1.

5.3.3 Height Analysis

Many applications for AFMs require measuring the heights of features and the features have one or more distinct levels. It is critical that the image be properly leveled to obtain reliable and accurate step height measurements.

Figure 5-8 A is an example of measuring heights from a histogram. Histogram measurements are the most accurate because the histogram is comprised of data from the entire image, which provides an averaging effect.

Figure 5-8 B shows one of the pitfalls of measuring step heights from a line profile. If only a single point is selected at the bottom of the profile and at the top of the profile, the step height value can vary dramatically. The error is a result of either the surface texture of the sample or noise in the image. This error may be minimized by averaging several lines that are in sequence in the image.

Figure 5-8 C illustrates a step height measurement technique where the average signal at the top and bottom of the profile are used. This technique is typically used when the type of sample under analysis is well characterized.

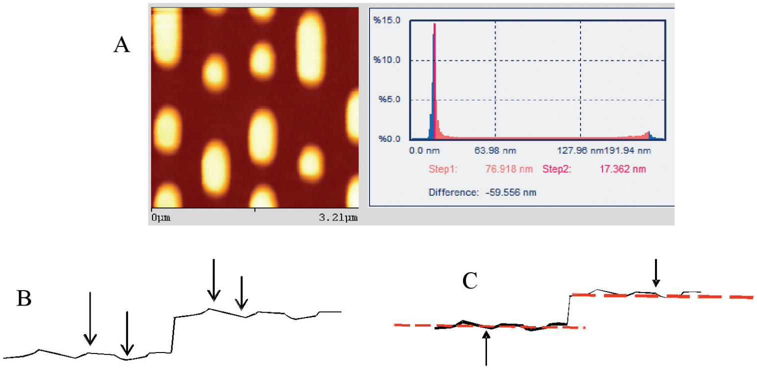


FIGURE 5-8 Three methods for measuring a step height:
A) Histogram Analysis
B) Single point at the top and bottom of a profile
C) A line fit to the top and bottom of the profile.

5.3.4 Particle Analysis

An AFM is capable of measuring nanoparticle images, making analysis of the nanoparticles possible. After an image of nanoparticles is properly leveled, it is possible to identify the particles in the image and then analyze the nanoparticles.

Nanoparticle Recognition

Auto-Detect: This method has a software algorithm that searches for height transitions associated with particles in the AFM image. This technique is ideal if the nanoparticles in the AFM image are well defined. It is advantageous because it requires little or no expertise from the operator.

Threshold Method: In the threshold method, the particles are identified in an image by settings established by the person making the analysis. A "threshold" in the image is selected so that the nanoparticles are above the threshold. A color scale is used in the image to visually facilitate setting the threshold (see Figure 5-9).

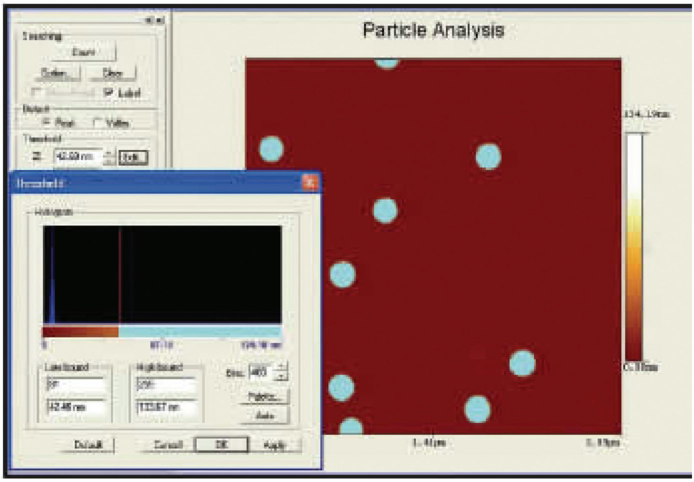


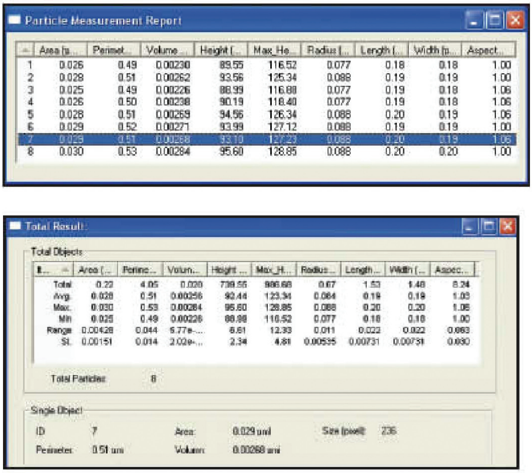
FIGURE 5-9 Nanoparticles on a surface are identified with software that searches for thresholds, or transitions in the image. The criteria for a threshold can be set manually or be automated. Once the particles in the image are identified they can be counted and then measurements may be made on each particle.

Count / Measure

Once the nanoparticles in the AFM image are identified they are automatically counted. The software typically does not count "partial" particles that are clipped at the edge of an image. However there is an option to enable counting partially measured particles. Each particle is assigned an individual number (see Figure 5-10).

Parameters that are automatically measured for each particle and presented in a tabular format include:

- Volume
- Area
- Length
- Height
- Aspect ratio
- Width
- Radius



Particle Measurement Report

	Area (μ...	Perimet...	Volume ...	Height (...	Max_He...	Radius (...	Length (...	Width (μ...	Aspect...
1	0.026	0.49	0.00230	89.55	116.52	0.077	0.18	0.18	1.00
2	0.028	0.51	0.00262	93.56	125.34	0.088	0.19	0.19	1.00
3	0.025	0.49	0.00226	88.99	116.88	0.077	0.19	0.18	1.06
4	0.026	0.50	0.00238	90.19	118.40	0.077	0.19	0.18	1.06
5	0.028	0.51	0.00269	94.56	126.34	0.088	0.20	0.19	1.06
6	0.029	0.52	0.00271	93.99	127.12	0.088	0.19	0.19	1.00
7	0.029	0.51	0.00268	93.10	127.23	0.088	0.20	0.19	1.06
8	0.030	0.53	0.00284	95.60	128.85	0.088	0.20	0.20	1.00

Total Result:

Total Objects

I...	Area (...	Perime...	Volum...	Height ...	Max_H...	Radius...	Length...	Width (...	Aspec...
Total	0.22	4.05	0.020	739.55	986.68	0.67	1.53	1.48	8.24
Avg.	0.028	0.51	0.00256	92.44	123.34	0.084	0.19	0.19	1.03
Max.	0.030	0.53	0.00284	95.60	128.85	0.088	0.20	0.20	1.06
Min	0.025	0.49	0.00226	88.98	116.52	0.077	0.18	0.18	1.00
Range	0.00428	0.044	5.77e-...	6.61	12.33	0.011	0.022	0.022	0.063
St.	0.00151	0.014	2.02e-...	2.34	4.81	0.00535	0.00731	0.00731	0.030

Total Particles: 8

Single Object

ID 7 Area: 0.029 um² Size (pixel): 236

Perimeter: 0.51 um Volume: 0.00268 um³

FIGURE 5-10 The critical parameters associated with each of the particles is presented in a table. Also, if there are several particles, the averages of each of the parameters are calculated and presented in a tabular format. The calculated values all have the same systematic errors associated with probe geometry.

Besides being able to analyze a singe particle, graphs may be created that show the distribution of particles as a function of any of the measured variables. Figure 5-11 shows the distribution of particles in an image versus the particle volume, volume vs height, and volume vs aspect ratio.

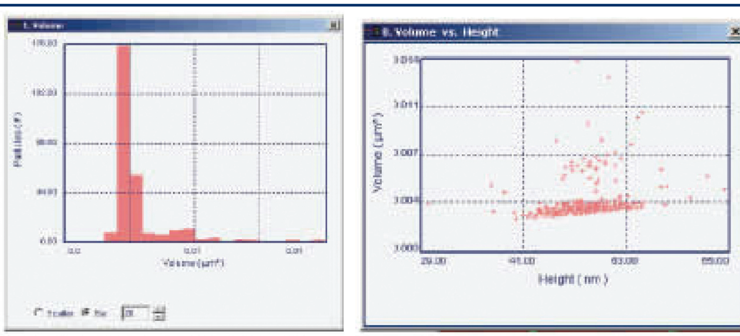


FIGURE 5-11 Graphs of the particle parameters may be created. Left: Plot of the number of particles with a specific volume. Right: Plot of particle volume versus particle height.

5.3.5 Grains Analysis

An AFM is an exceptional tool for measuring grain structure on surfaces. This is because the AFM has great contrast on flat samples. The analysis of AFM images of grains is advantageous because the images have three dimensional topography and the grain boundaries can be easily identified. Figure 5-12 illustrates the use of software for identifying grains of polysilicon.

After the grains are identified with software, the volume, size, area, etc., can be calculated and displayed in a tabular or graphical format.

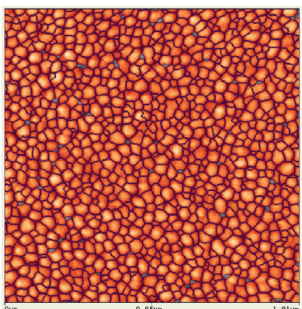


FIGURE 5-12 Grains may be identified with thresholding software. Analysis of grains is very similar to the analysis of particles

5.3.6 Technical Samples

An AFM is helpful with product development and quality control of many high technology products such as DVDs. Software is available for automatically analyzing such samples (see Figure 5-13).

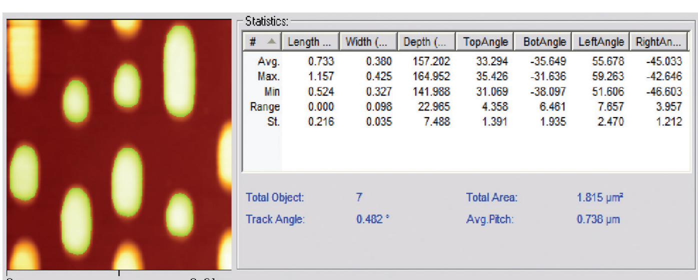


FIGURE 5-13 Using thresholding software, analysis of technical samples such as DVD bits can be automated.

CHAPTER 6

AFM Image Artifacts

All measurement instrumentation used by scientists and engineers for research, development and quality control generates results that may have artifacts. This chapter serves as a guide to identifying common artifacts that occur in AFM images. This guide is organized in sections that are divided by the sources that generate the image artifacts.

There are four primary sources of artifacts in images measured with atomic force microscopes. They are:

- Probes
- Image Processing
- Scanners
- Vibrations

6.1 Probe Artifacts

Images measured with an atomic force microscope are always a convolution of the probe geometry and the shape of the features being imaged. If the probe is much smaller than the features of the images being measured, then the probe-generated artifacts will be minimal and the dimensional measurements derived from the images will be accurate.

Avoiding artifacts from probes is achieved by using the optimal probe for the application. For example, if the features that are being imaged have feature sizes of interest in the 100 nanometer range, a probe as large as 10 nanometers in diameter will be adequate for getting good images with no artifacts. In some cases, even if the probe is not as sharp as the object being imaged, it is still possible to get accurate information from the image.

Common artifacts are:

- Features on a surface appear too large
- Features in an image appear too small
- Strangely shaped objects
- Repeating strange patterns in an image

6.1.1 Features on a Surface Appear Too Large

Often the size of features on the surface such as nanotubes or nanospheres look larger than expected. However, the height of the feature when measured by a line profile is correct.

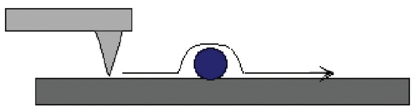


FIGURE 6-1 Motion of an AFM probe as it goes over a sphere that is attached to a surface. In such a measurement the side of the probe will cause a broadening of features in the image.

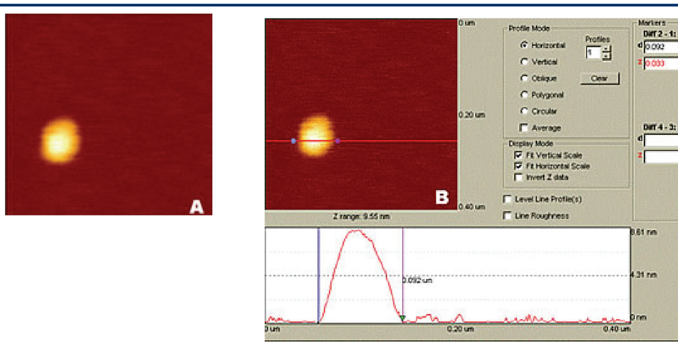


FIGURE 6-2 A-B: 400 × 400 nm AFM image of an 8 nm diameter sphere. A: The line profile of the image shows a diameter of 92 nm and a height of 8 nm. B: The broadening in the image is caused by the shape of the probe used for measuring this AFM image.

6.1.2 Features in an Image Appear Too Small

If the probe needs to go into a feature that is below the surface, the size of the feature can appear too small. The line profile in these cases is established by the geometry of the probe and not the geometry of the sample. However, it is still possible to measure the opening of the hole from this type of image. Also, the pitch of repeating patterns can be accurately measured with probes that don't reach the bottom of the features being imaged.

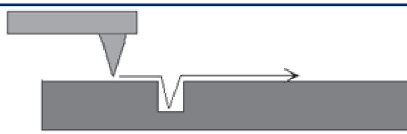


FIGURE 6-3 The motion of an AFM probe as it moves over a hole in a surface. Because of the width of the probe, it does not reach the bottom of the hole.

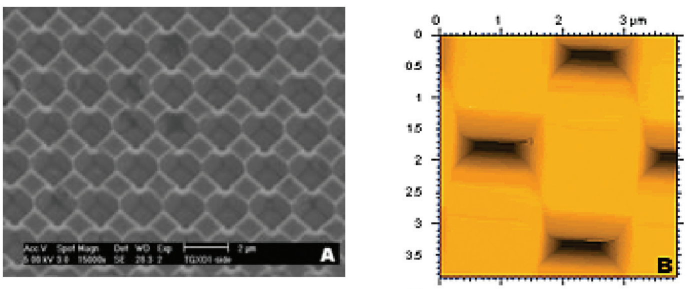


FIGURE 6-4 A: Scanning electron microscope image of a test pattern of squares (NT-MDT TXO1). The sides of the squares are all equal. B: AFM image of the test pattern. Because the probe is not sharp, the test pattern squares appear much smaller than they should. The features in the AFM image appear as rectangles and not as squares.

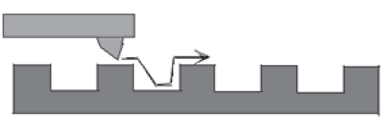


FIGURE 6-5 This "chipped" AFM probe follows the geometry of the sample surface and creates an image with a substantial artifact.

6.1.3 Strangely Shaped Objects

If the probe gets broken or chipped before an image is measured, strangely shaped objects may be observed that are difficult to explain. For example, when scanning a semiconductor test pattern, it can appear as though the tip is at a large angle to the surface (as described in Section 6.2.1). However, the probe to sample angle would have to be extreme to explain the image artifact (see Figure 6.6).

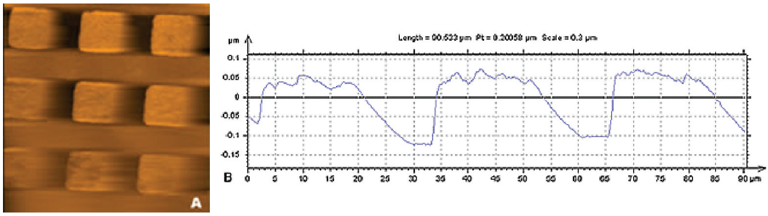


FIGURE 6-6 A: This AFM image of a test pattern appears to have dark right edges. B: The artifact can be easily seen in the line profile. Although this artifact could be explained by a large angle between the probe and surface, the probe surface angle cannot be this large. Scan size: 91µm X 91µm.

6.1.4 Repeating Strange Patterns in an Image

If the features on a surface are much smaller than the probe, it is possible to see large numbers of repeating patterns in an image. The patterns will often appear as triangles, especially if silicon probes are used for imaging. Example: Images of colloidal gold particles reflect the shape of the tip rather than their own geometry. Compare the SEM images of tips and related AFM images of spheres in the Figure 6-7.

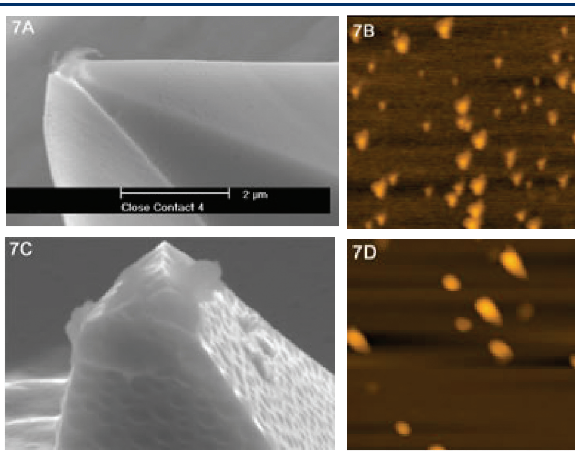


FIGURE 6-7 The AFM images at the right, B (5 nm in diameter) and D (28 nm in diameter), are of nanospheres that are supposed to be perfect spheres. At the right, A and C, are scanning electron microscope images of the AFM probes used for getting the images of the spheres. Because the chipped probes are much larger than the spheres, the AFM images reflect the probe's geometry. The scan size is 700nm × 700nm.

6.2 Scanner Artifacts

Scanners that move the probe in an atomic force microscope in the X, Y and Z directions are typically made from piezoelectric ceramics. As electromechanical transducers, piezoelectric ceramics are capable of moving a probe very small distances. However, when a linear voltage ramp is applied to piezoelectric ceramics, the ceramics move in a nonlinear motion. Further, the piezoelectric ceramics exhibit hysteresis effects caused by self-heating. Artifacts can also be introduced into images because of the geometry of the scanner. The positioning of the scanner relative to the sample can also create artifacts.

6.2.1 Probe / Sample Angle

If the features that are being imaged by the AFM are much larger in profile than the probe, and the image does not seem "correct", the artifact may be caused by a non-perpendicular probe surface angle. Ideally, the probe of the microscope should be perpendicular to the surface.

FIGURE 6-8 In this example the probe is much sharper than the feature it is scanning across and should give a correct image. However, because of the extreme probe sample angle, the line profile will show an artifact at the left edge of the feature.

Solving this problem is achieved by adjusting the angle between the probe and the sample so that they are perpendicular. In some microscopes the probe is designed to be at a 12 degree angle with respect to the sample. Also some AFM microscopes do not have mechanical adjustments to control the probe/sample angle.

6.2.2 X-Y Calibration / Linearity

All atomic force microscopes must be calibrated in the X-Y axis so that the images presented on the computer screen are accurate. The motion of the scanners must also be linear so that the distances measured from the images are accurate. With no correction, the features on an image will typically appear smaller on one side of the image than on the other.

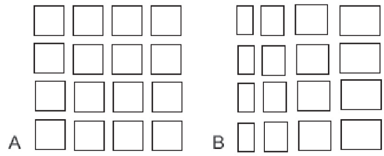


FIGURE 6-9 A test pattern with squares, A, will appear severely distorted if the piezoelectric scanner in the AFM is not linear as in B.

Once the scanner is properly linearized, it is also critical that the scanner be calibrated. For example, it is possible for the scanner to be linear but not calibrated. If the calibration is incorrect, then the X-Y values measured from line profiles will be incorrect.

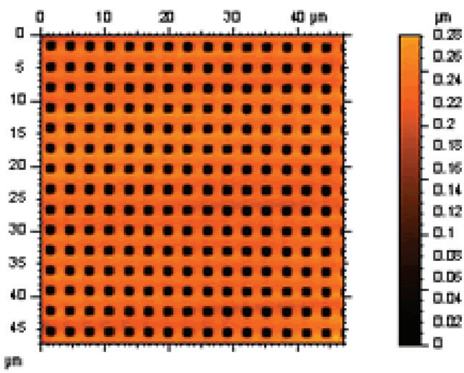


FIGURE 6-10 This AFM image of a test pattern is very linear. The spacing of the squares at the top, bottom, left and right sides are all the same distance apart. It appears as it should. A common method for correcting the problems of X-Y non-linearity and calibration is to add calibration sensors to the X-Y piezoelectric scanners. These sensors can be used to correct the linearity and the calibration in real time.

6.2.3 Z Calibration / Linearity

Height measurements in an AFM require that the piezoelectric ceramics in the Z axis of the microscope be both linear and calibrated. Often the microscope is calibrated at only one height. However, if the relationship between the measured Z height and the actual Z height is not linear, then the height measurements will not be correct.

FIGURE 6-11 This graph shows the relationship between an actual Z height and a measured Z height in an atomic force microscope. Often only one calibration point is measured as shown by the grey circle, and the Z ceramic is assumed to be linear, as shown by the blue line. However, as is often the case, the ceramic is nonlinear, as shown by the red line. In such cases incorrect Z heights are measured with the microscope unless the feature being measured is close to the calibration measurement.

6.2.4 Background Bow / Tilt

The piezoelectric scanners that move the probe in an atomic force microscope typically move the probe in a curved motion over the surface. The curved motion results in a "Bow" in the AFM image. Also, a large planar background or "Tilt" can be observed if the probe/sample angle is not perpendicular.

Often the images measured by the AFM include a background "Bow" and a background "Tilt" that are larger than the features of interest. In such cases the background must be subtracted from the image. This is often called "leveling" or "flattening" the image.

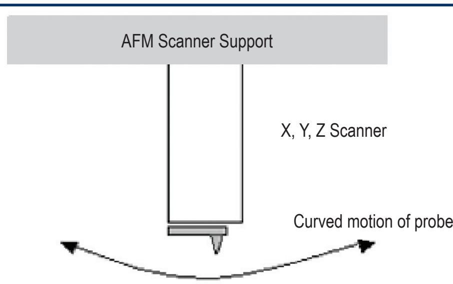


FIGURE 6-12 An AFM piezoelectric scanner is often supported at the top by a mechanical assembly. Thus the motion of the probe is nonlinear in the Z axis as it is scanned across a surface. The motion can be spherical or even parabolic depending on the type of piezoelectric scanner.

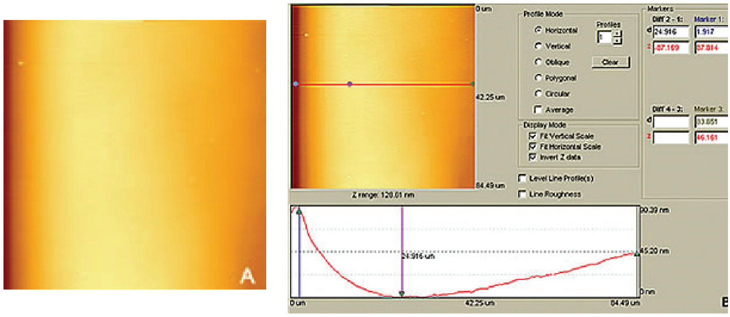


FIGURE 6-13 A: An 85 × 85 micron image of a flat piece of silicon. The bow introduced into the image is seen at the edges. B: A line profile across this image shows the magnitude of the bow.

6.2.5 Z Edge Overshoot

Hysteresis in the piezoelectric ceramic that moves the cantilever in the perpendicular motion to the surface can cause edge overshoot. This problem is most often observed when imaging micro-fabricated structures such as patterned Si wafers or compact discs. The effect can cause the images to be visually better because the edges appear sharper. However, a line profile of the structure shows errors.

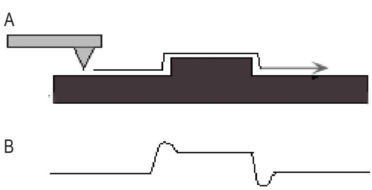


FIGURE 6-14
A: The probe is scanned from left to right across a feature on a surface.
B: Overshoot may be observed in the line profile at the leading and trailing edge of the structure.

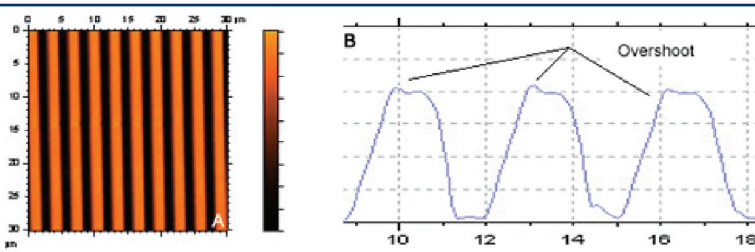


FIGURE 6-15 A: The AFM image of a test pattern appears to have no artifacts. B: However, a line profile of the test pattern shows overshoot at the top of each of the lines.

6.2.6 Scanner Drift

Drift in AFM images can occur because of "creep" in the piezoelectric scanner and because an AFM can be susceptible to external temperature changes. The most common type of drift occurs at the beginning of a scan of a zoomed-in region of an image. This artifact causes the initial part of a scan range to appear distorted. Drift artifacts are most easily observed when imaging test patterns. Drift will cause lines that should appear straight to have curvature.

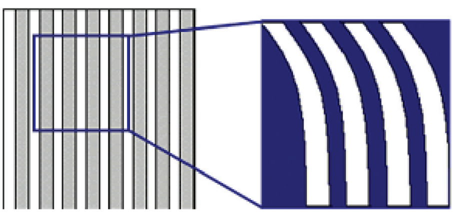


FIGURE 6-16 After a region of a sample is scanned with the AFM it is common to "zoom" into a small section of the image to get a higher magnification of an image. Scanner drift will cause the image to appear distorted at the beginning of the scan.

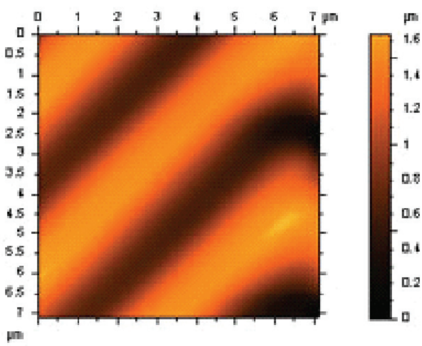


FIGURE 6-17 Zoomed image showing a distortion at the beginning of the scan. The scan angle is 45 degrees.

6.2.7 X-Y Angle Measurements

If the motion generated by the X-Y scanner is not orthogonal, then there can be errors in the horizontal measurements in an image. This error, or artifact, can best be seen when imaging a test pattern with squares. The error in orthogonality can be measured by using a straight edge to measure "orthogonal" lines in the images.

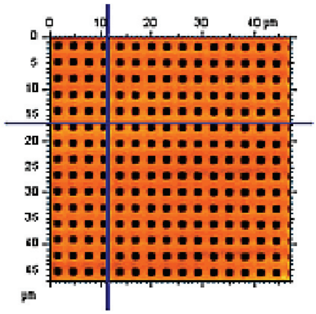


FIGURE 6-18 The blue lines drawn on this image show that the scanner has no measurable cross-talk between the X and the Y axis. The lines are orthogonal.

6.2.8 Z Angle Measurements

Mechanical coupling between the piezoelectric ceramics that move the probe in the X or Y directions and the Z direction can cause substantial errors when trying to measure side wall angles with the AFM. This error can best be measured with a sample that has repeating triangle structures.

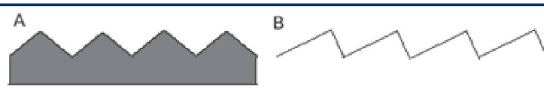


FIGURE 6-19 A-B: Image A: This cross section is an ideal sample for demonstrating the ability of an AFM to measure angles. The sample has a series of repeating triangles at its surface. Image B: A line profile of the sample shows that the triangles do not appear symmetric.

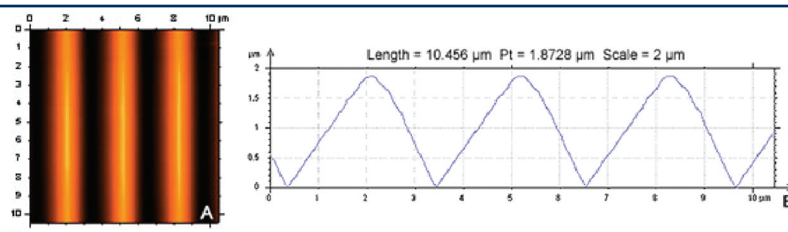


FIGURE 6-20 A: The AFM image of a sample having a triangle pattern at its surface. B: A line profile extracted from the AFM image.

6.3 Image Processing

Image processing is required before viewing or analyzing almost all AFM images. Most AFM products are supplied with very powerful image display and analysis software. Properly used, the image processing software will typically not introduce artifacts into an image. This section presents some of the common artifacts that can be introduced into AFM images by the image processing software.

6.3.1 Leveling

As mentioned in Section 6.2.4, most images have some tilt and bow that is introduced to the images by the scanner or stage configuration. There are a number of background subtraction options that are possible. The two most common types are:

- Line by line leveling - 0 to 4(th) order
- Plane Leveling - 0 to 4(th) order

Also, software typically allows you to exclude areas from the leveling. When an area is excluded, it is not used for the calculation of the background in the image.

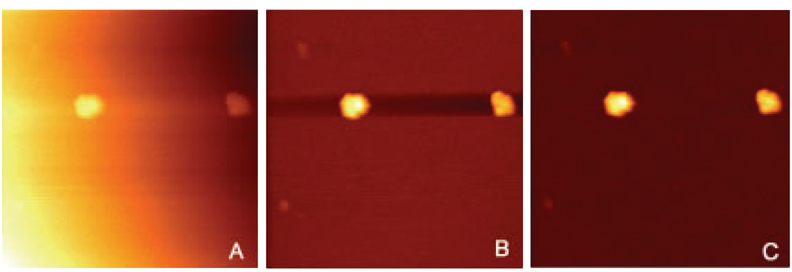


FIGURE 6-21 A-C: AFM images of a 1.6 × 1.6 micron area of nanospheres on a surface. A: The original image measured by the AFM before any image processing. Tilt is easily recognized in the image as the right side of the image appears darker than the left side of the image. B: The AFM image shown in "A" after a line-by-line leveling of the image with a first order background correction. The dark band in the image is caused by the image processing and is not a real structure. C: Particles are excluded from the background subtraction process to derive this image.

6.3.2 Low Pass Filter

A low pass filter is often used to "smooth" data before it displays. Such filters can cause steps in images to appear distorted.

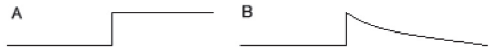


FIGURE 6-22 Low pass filtering of the step on the left (A) results in the shape shown on the right (B). The amount of distortion depends on the amount of filtering applied to the image.

When images are viewed that have substantial low pass filtering, the dimensions in the image can appear distorted. Other artifacts can appear as a sharpness at the edge of steps in an image.

6.3.3 Matrix Filter / Smoothing

Matrix filtering is very effective at "smoothing" images and removing noise from the image. However, the filtering process often reduces the resolution of the image. As a rule of thumb, if the image has no noise in it, then the data has probably been compromised.

6.3.4 Fourier Filtering

Periodic structures can easily be introduced into images with Fourier filtering. This can be used for creating "atomic structure" in images. As an example, images of "white noise" can be filtered to give periodic structure that looks like atomic structure.

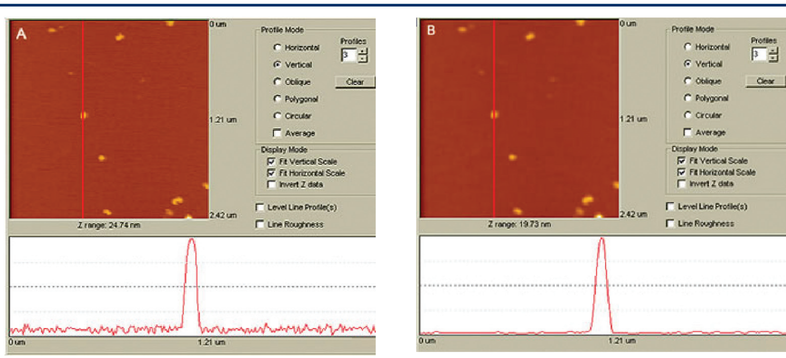


FIGURE 6-23 A: AFM image of nanospheres with no filtering. The image shows noise in the associated line profile. B: The image generated after matrix smoothing. The line profile shows no noticeable noise and the shape of the particle is altered.

6.3.5 Image Looks Too Good

If an AFM image looks too good to be true it probably is. All measurement techniques have some noise associated with them. Because AFM data is completely electronic, it is possible to take an image and alter it with image enhancement techniques to create a beautiful picture that does not represent the structure of the surface.

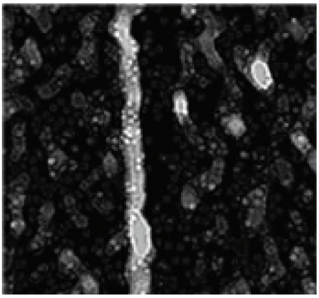


FIGURE 6-24 This 850 × 850 nm image of a nanotube had substantial noise when originally measured. Filtering added the “nodules” to the image making it seem like a much higher resolution image.

6.4 Vibrations

Environmental vibrations in the room where the AFM is located can cause the probe in the microscope to vibrate and make artifacts in an image. Typically, the artifacts appear as oscillations in the image. Both acoustic and floor vibrations can excite vibrational modes in an AFM and cause artifacts.

6.4.1 Floor Vibrations

Often, the floor in a building can vibrate up and down several microns at frequencies below 5 Hz. The floor vibrations, if not properly filtered, can cause periodic structure in an image. This type of artifact is most often noticed when imaging very flat samples. Sometimes the vibrations can be started by an external event such as an elevator in motion, a train going by, or even people walking in a hallway.

6.4.2 Acoustic Vibrations

Sound waves can cause artifacts in AFM images. The source of the sound can be from an airplane going over a building or from the tones in a person’s voice. Below is an image that shows the noise derived from a person talking in the same room as the microscope.

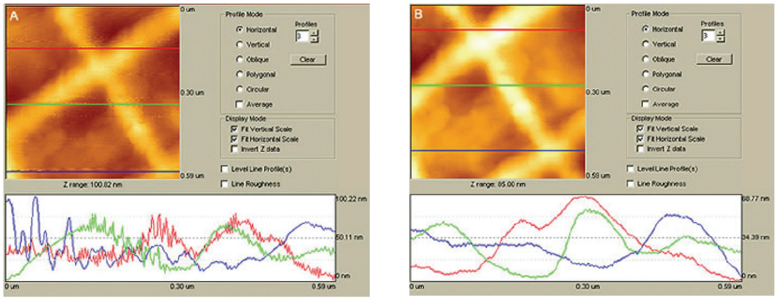


FIGURE 6-25 This high resolution image of a test grid shows the effect of acoustic noise on an image. A: Image and line profiles measured while acoustic noise was present in the room. B: Image that was measured without the acoustic noise.

6.5 Other Sources

6.5.1 Surface Contamination

Substantial contamination at the surface of a sample such as a fingerprint or oil film can cause AFM image artifacts. Such artifacts appear as streaks on the image especially in locations where there are “sharp” features and edges on the sample’s surface. Often the streaking can be reduced or even eliminated by cleaning the sample with a high purity solvent.

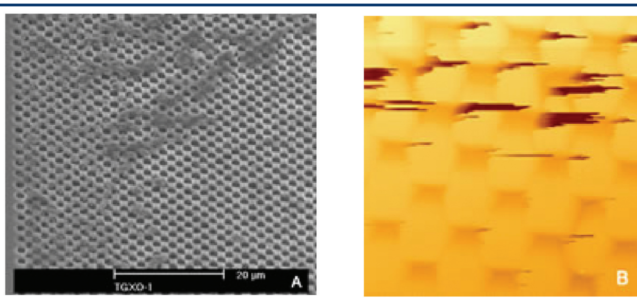


FIGURE 6-26 A: SEM image of a test pattern that is contaminated. B: AFM image of the same test pattern that is covered with contamination. The contamination is identified by the streak marks at the top of the scan.

6.5.2 Electronics

Image artifacts can appear in AFM scans because of faulty electronics.

Artifacts from electronics most often appear as oscillations or unexplainable repeating patterns in an image. Electronic ground loops and broken components are usually the source of electronic noise.

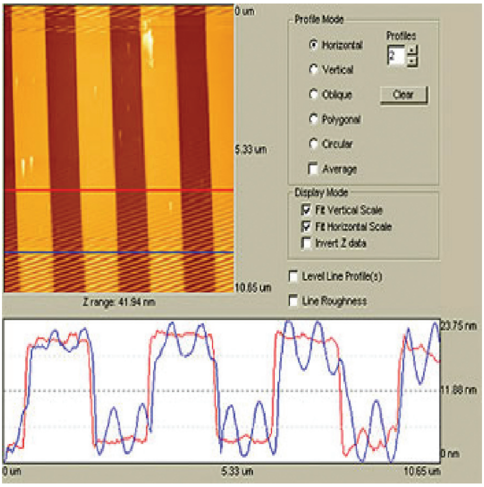


FIGURE 6-27 Image of a test pattern that has electronic noise at the top and bottom of the scan. The electronic noise in this case was a result of not having a ground wire attached to the stage. The artifact is identified by the oscillations.

6.5.3 Vacuum Leaks

Atomic force microscopes that are designed for imaging wafers and discs often use a vacuum chuck to hold the wafer/disc while scanning images. A leak in the vacuum between the specimen holder and the specimen can cause image artifacts. The artifact causes a loss of resolution in the image. Cleaning the vacuum chuck and sample often eliminates this problem.

6.5.4 PID Settings / Scan Rate

If the PID Settings used while scanning are not optimized, there is a potential for an artifact being generated in an image. This is because the probe is not tracking the surface, and the cantilever is bending to pass over surface features. This artifact can be identified easily by monitoring the error signal. If the error signal is too large, then the probe is not correctly tracking the surface.

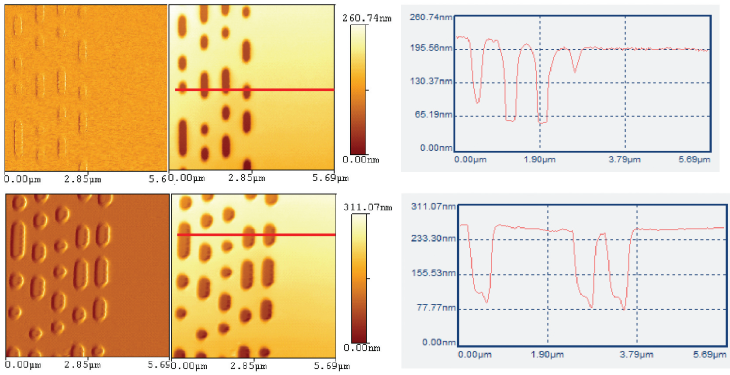


FIGURE 6-28 Example of an artifact created by not having the PID parameters optimized while scanning. In the upper image parameters are optimized, in the lower image parameter are not optimized and the error signal is large.

6.5.5 Laser Interference Patterns

Interference patterns can be created by the laser used in the LL-AFM sensor. The interferences appear as low frequency background oscillations in images and typically have a period that is similar to the wavelength of the laser light being used in the AFM scanner. The interferences can be created from patterns on the sample's surface, or from interferences in the cantilever. Figure 6-29 illustrates this type of artifact.

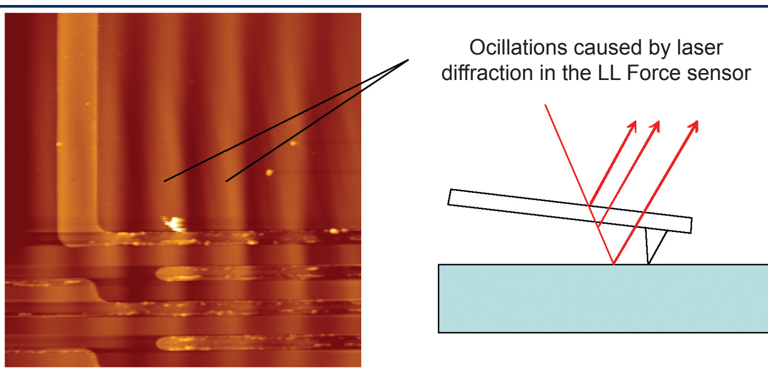


FIGURE 6-29 Wavy patterns (oscillations) in this image are an artifact created by laser diffraction with the sample's surface features. Scanning in a non-patterned area gives images with no observable oscillations.

CHAPTER 7

AFM Applications

Because the AFM is capable of measuring nanometer scale images of insulating surfaces with little or no sample preparation, it has a vast number of applications in many areas of science and technology. This chapter serves as an introduction, or a snapshot, to AFM applications. The applications are presented in a “picture” format. Besides the applications listed in this chapter, Chapter 4 on Imaging Modes also presents several applications for atomic force microscopes.

The primary uses for the AFM are:

- Visualization: The AFM measures three dimensional images of surfaces and is very helpful for visualizing surface topography.

- Spatial Metrology : Nanometer sized dimensions of surface features are measurable with the AFM.

- Physical Property Maps: With many of the modes (see Chapter 4) it is possible to measure surface physical property maps. These techniques are, for the most part, qualitative.

After the initial invention of the AFM in 1986 there was a great effort focused on developing AFM instrumentation. Within a few short years, the instruments moved from being esoteric devices requiring a Ph.D. to operate to table top instruments that could be operated by technicians. From 1990 to 2000 applications for the AFM moved from fundamental physics to most areas of science and technology. It is estimated that in 2006 there are approximately 10,000 AFM’s in use around the world.

As with most scientific instruments, the initial applications for the AFM were in basic and applied research; that is, the AFM was used to push forward the frontiers of knowledge. The primary objective of this type of research is to publish a paper in a scientific or technical journal.

After its initial use for basic research, the AFM began to be used for product and process development. The goal of this type of application is to create a better product. Many of these applications are in the high and advanced technology industries such as the disk drive and semiconductor industries.

It is expected that as nanotechnology processes become important for producing products, the AFM will find many applications in quality and process control. Again, the initial process control applications for the AFM are in the semiconductor and data storage industries.

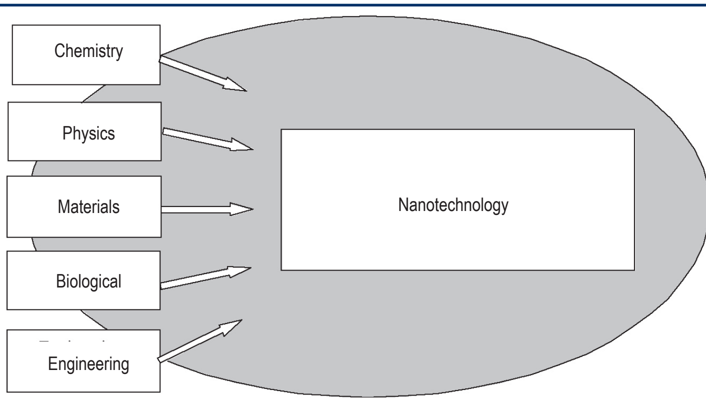


FIGURE 7-1 Several traditional scientific and engineering disciplines contribute to the field of nanotechnology.

Beginning in approximately 2000, the emergence of nanotechnology funding throughout the world led to new applications for atomic force microscopes. In many cases, nanotechnology is directly related to the traditional fields in science and engineering. Figure 7-1 shows the relationship between traditional science and technology and nanotechnology.

There are three critical issues that must be addressed to successfully measure AFM images for all applications. They are:

a) Select the optimal probe for the application – The probe must be sharp enough to image the features of interest, and must have a shape such that the measurements are accurate if required.

b) Prepare the sample correctly – The sample must be rigidly mounted in the microscope and materials placed on a surface must be rigidly attached to the surface.

c) Make sure to use the correct mode and scan parameters for the application – Topography images may be measured in contact or vibrating mode depending on the hardness of the sample and the resolution that is required.

In the following sections, applications in several areas of science and technology are presented. In many cases, an entire book would be required to cover all of the detailed information associated with the application. In most cases, an image is presented to illustrate the application.

7.1 Physical Science

AFM is rapidly becoming a standard microscopy technique for visualizing and measuring a material's surface structure in the physical sciences. The types of structures that are scanned with the AFM include: surfaces of bulk materials, thin films, and nanostructures that are located on a surface. There are a large number of materials that may be imaged with an AFM, including polymers, ceramics, metals, crystals, and minerals. The scan ranges for imaging in the physical sciences range from a few nanometers all the way up to tens of microns.

7.1.1 Polymer Composites

Since they are electrically insulating, measuring high resolution images of composites is very difficult with an SEM/TEM because the samples must be coated with a conductive layer. An AFM can readily measure images of composite polymers with little or no sample preparation. With techniques such as vibrating phase mode (Section 4.3.2) it is possible to visualize differences in the composition in composite polymers. The image in Figure 7-2 shows the changes in mechanical properties of a polymer material with phase imaging mode. The distribution of materials in the polymer matrix is clearly observed.

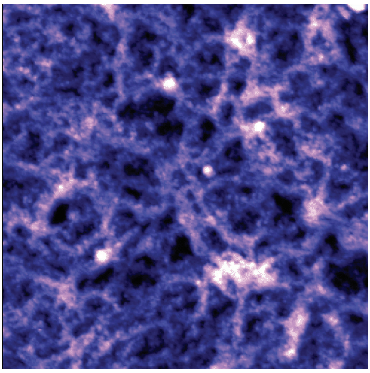


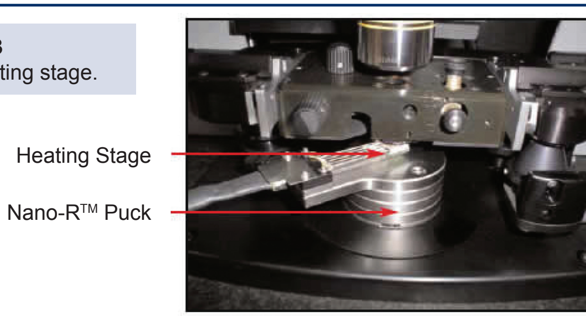
FIGURE 7-2 Polymer – Carbon Composite Film 50 X 50 u.

7.1.2 Phase Transitions

As materials undergo phase transitions, they often have changes in their surface structure that can be readily imaged with an AFM. There are many methods for heating a sample including:

a) on a heating stage external to the AFM
b) on a heating stage internal to the AFM
c) with a small heater located at the end of the AFM probe

FIGURE 7-3
Sample heating stage.



7.1.3 Surface Texture

Atomic force microscopes give exceptional contrast on samples with little or no surface features. Such contrast is not possible with any other type of microscope. It is critical that the viewing area of the image is on the same length scale as the features giving rise to the surface texture. As an example, the image shown here is of a 1 X 1 micron region of a silicon wafer. The surface roughness of this area is 0.1 nm. If there are larger scale surface features, such as waviness or bow in the wafer, then the AFM will not measure this surface topography.

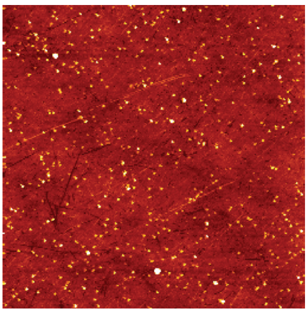


FIGURE 7-4 Polished Quartz 10 X 10 u.

7.1.4 Defects

Defects in many types of materials such as metals, crystals, and ceramics are easily measured with an AFM. Because the AFM measures three dimensional surface structure, it is possible to measure not only the area of the defect but also the volume of a defect at a surface. A drawback of using an AFM for imaging defects is that the field of view of an AFM is fairly small, so the density of the defects must be such that at least one defect is in the FOV. This problem is sometimes dealt with by using another microscope or instrument for locating the defect. The AFM is then used to fully characterize the defect.

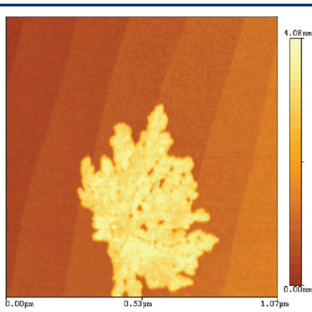


FIGURE 7-5 Defect on surface of sapphire terraces.

7.1.5 Crack / Scratch Propagation

An AFM is ideal for studying crack propagation in surfaces because the AFM gives great contrast on flat samples. If the crack is longer than the FOV of the AFM scanner, it may be necessary to measure several AFM images in succession. Measuring the images can be facilitated if the AFM stage has a motorized stage that can be programmed to measure the entire crack. Also, it is often possible to place a device for creating stresses and strains in materials directly in the AFM stage. In such a case, direct images of crack formation are measurable.

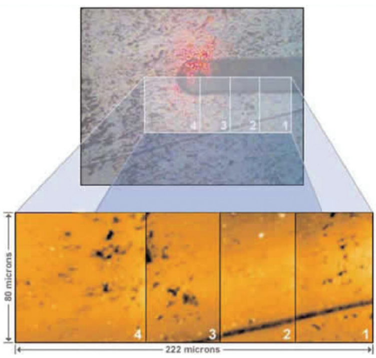


FIGURE 7-6 Top: optical microscope image of AFM cantilever near scratch. Bottom: AFM image of scratch, stitched form 4 images.

7.1.6 Coatings

Many types of coatings on surfaces are directly measurable with an AFM. The only requirement is that the coating surface roughness be less than the dynamic range of the Z piezo in the AFM. The thickness of coating can be measured with a cross section or by having a region of the sample surface that has a transition to the coating.

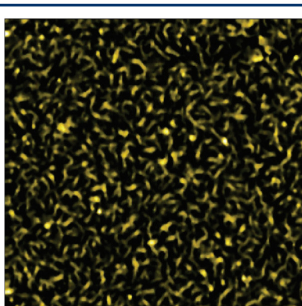


FIGURE 7-7 Polypeptide Coating 4.8 X 4.8 u.

7.1.7 Nanoparticles

The AFM can easily visualize nanoparticles with sizes ranging from a few nanometers to a few microns. It is possible to measure the size of individual nanoparticles as well as measure the parameter distribution of an ensemble of nanoparticles. Parameters such as particle size, volume, circumference and surface area are readily measured. One of the greatest challenges to measuring nanoparticles is developing methods for distributing the nanoparticles on a surface.

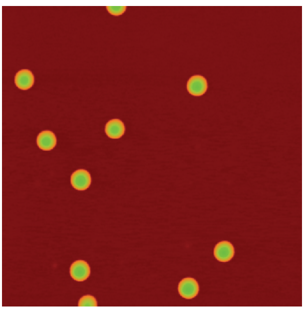


FIGURE 7-8 AFM image of 100nm nanoparticles 2.8 u X 2.8 u.

7.1.8 Carbon Nanotubes

High resolution images of both single wall and multi-wall carbon nanotubes are measurable with the AFM. The nanotubes must be dispersed on a flat surface for imaging.

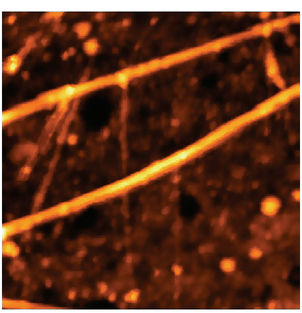


FIGURE 7-9 AFM image of single and multi-wall CNT.

7.1.9 Crystal Structure

Atomic terraces on crystal surfaces are readily measured with an AFM in ambient air. It is not possible, however, to measure the topography of single atoms at the surface of crystals. This is because the effective diameter of the probe is increased dramatically by the contamination layer on surfaces in ambient air. It is possible, however, to measure atomic structure with an AFM in an ultra high vacuum environment.

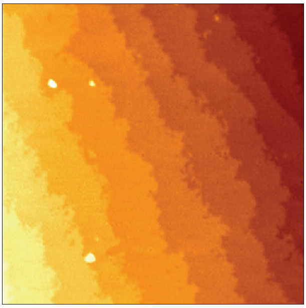


FIGURE 7-10 Atomic terraces on Strontium Titanate.

7.2 Life Sciences

The AFM has great promise for measuring surface structure of biological material. For example, the AFM is the only microscopy technique that allows making nanometer scale images with the sample submerged in liquid.

7.2.1 Cells

An AFM can readily measure images of cells both in ambient air and submerged in a liquid. The cells must be rigidly adhered to a surface for successful imaging. With the AFM it is possible to measure the mechanical activity of a cell by simply placing the probe on the surface of the cell and monitoring the motion of the AFM cantilever. An advantage of the AFM for imaging cells is that the cell does not have to be coated, and in fact can still be alive when imaged.

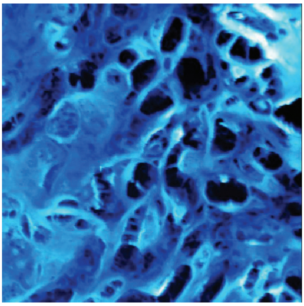


FIGURE 7-11 Breast Cancer Cells 78 X 78 microns.

7.2.2 Bio-Molecules

AFM images of bio-molecules such as DNA are easily measured as long as the bio-molecules are directly attached to a surface. Activated substrates are available for creating DNA images with an AFM. There are many examples of AFM DNA images as well as many images of other types of bio-molecules such as proteins. The AFM is capable of systematically removing genetic material from DNA or chromosomes by scratching the surface of the material.

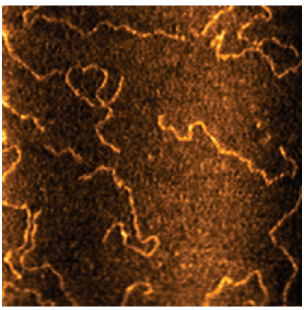


FIGURE 7-12 DNA image 1.2u x 1.2u.

7.3 High Technology

The high technology industries, including semiconductors, data storage and advanced optical devices, use AFM for product/process development and in some cases for process control. Many of these industries already rely on manufacturing procedures that create structures with nanometer sized dimensions.

7.3.1 Semiconductor

There are numerous applications for the AFM in the semiconductor industry both in the front and back end. The AFM has played a critical role in the development of many semiconductor processes such as CMP in the mid 1990's. The greatest limitation for the AFM in applications for the semiconductor industry is the availability of reliable probes with nanometer scale dimensions. Advanced applications include: electrical testing, etch verification, and secondary defect review.

Because of probe geometry, not all dimensions of device structures are measurable with an AFM. In Figure 7-13, dimensions that are measurable include:

H:	Measured
P:	Measured
W1:	Possible
W2:	Difficult – special probes

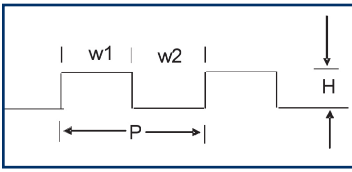


FIGURE 7-13 Illustration of device structure.

Figure 7-14 illustrates the use of AFM for measuring the angles of a side wall in a trench. This trench is relatively large, and because the wall angle is less than the probe angle, the measurement is accurate. Measurement of smaller structures is dependent on the availability of probes with optimized geometries.

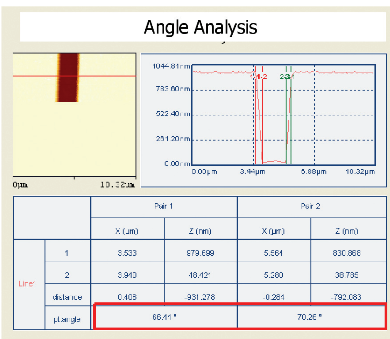


FIGURE 7-14 AFM measurements on a trench structure.

7.3.2 Data Storage

Data storage requires creating structures with nanometer-sized dimensions. The AFM is utilized for product development of both magnetic and optical mass storage devices. Applications in the magnetic storage area include the visualization of magnetic bits, pole tip recession, and platter surface texture.

Bits and tracks in DVD and CD-R/W storage media are visualized with an AFM. DVD bit metrology measurements can yield all of the critical dimensions of a bit as well as track dimensions. Specialized software is available for automatically calculating the critical dimensions of DVD bits. The software identifies the bits and then uses specialized algorithms for calculating the bit's dimensions. Tables and graphs of the dimensional distributions can be created.

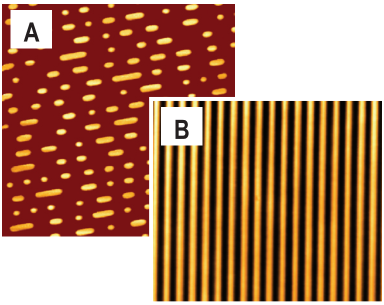


FIGURE 7-15 A: DVD bits 11.7 x 11.7 u, B: CD R/W 23 x 23 u.

7.3.3 Advanced Optical

Optics play a critical role in many high technology products such as cameras, video recorders, and flat panel displays. The AFM can be very helpful for metrological measurements when optical profilers cannot be used because the specimen under study is transparent.

Micro-Optics

Micro-optical devices are often created from insulating material and cannot be viewed in an SEM/TEM. Further, because they are transparent, an optical profiler does not give accurate metrological measurements. The AFM however, is a mechanical imaging tool and does, in fact provide accurate metrological measurements on optically transparent materials.

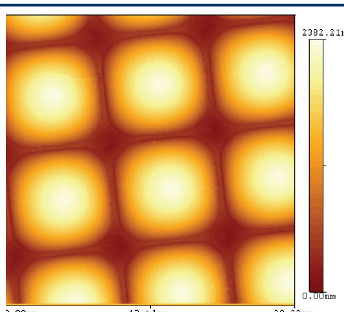


FIGURE 7-16 AFM image of a micro lens.

LCD Display

The AFM is ideal for making measurements on LCD screens and components. Figure 7-17 illustrates the AFM image of steps in an LCD screen that are between 2 and 50 nm. Additionally the nanoparticle structure on the surface of the LCD screen is readily visualized.

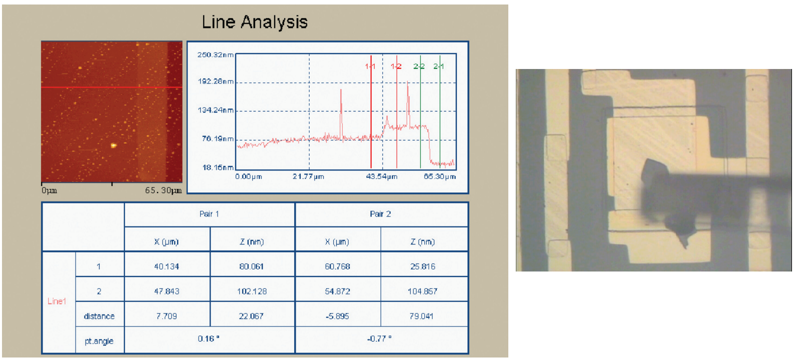


FIGURE 7-17 Line profile analysis of LCD structures.

7.4 Industrial

Many traditional industries are now finding that products can be modified and improved using nanometer-sized components. Often the industrial processes used to create products already include nanoscale components. Industries that are presently developing or using nanoscale components include the health care, paper, automotive, cosmetic, and aerospace industries.

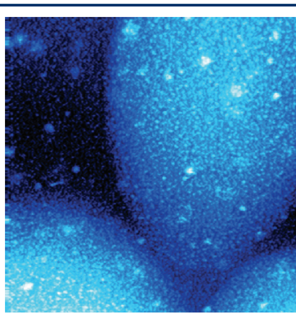


FIGURE 7-18 AFM image of ink drops deposited on paper.

Paper

Paper coatings are often comprised of nanoparticles. The size, material, and distribution effect the gloss of the paper. An AFM is ideal for visualizing and characterizing paper coatings.