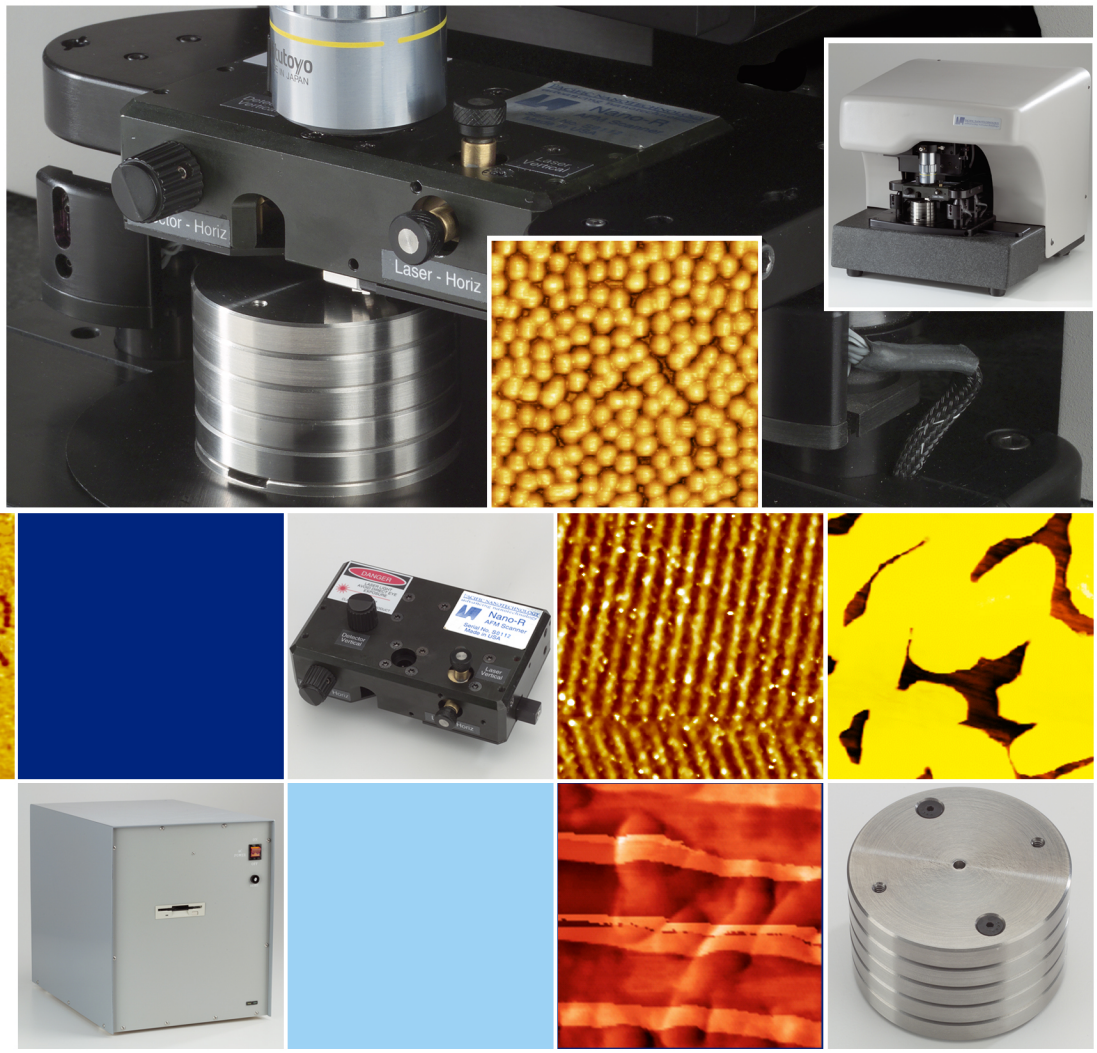
Nano-R™ AFM

User's Manual



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advancing nanotechnology

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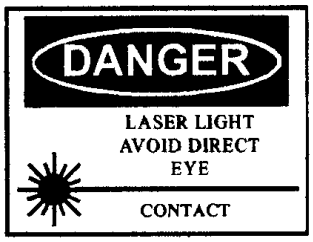
LASER OPERATION: AFM SCANNING HEAD LASER



WARNING: NEVER LOOK DIRECTLY INTO THE LASER BEAM

IN ORDER TO AVOID THE POSSIBILITY OF THE USER INADVERTENTLY LOOKING INTO THE LASER, ALWAYS USE THE SOFTWARE OR HARDWARE TO SWITCH THE LASER OFF BEFORE RAISING THE HEAD TO EYE LEVEL.

The diode laser in the Nano-R™ scanning head complies with US 21 CFR 1040.10 and is certified as a Class IIIa laser. The laser wavelength is 670nm and the maximum power is 3 mW.

In addition to the above, please follow laser safety control measures in American National Standards Institute Z136.1-1986.

HIGH VOLTAGE

Wherever high voltage is present on the system, extreme care should always be taken to avoid direct contact while the instrument hardware is powered on. Always power off the equipment before attempting to remove any panels or PC boards and before touching any connectors by hand or with electrically conductive tools.

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Preface

INTRODUCTION

The Nano-R™ AFM is an easy-to-use, high-performance atomic force microscope (AFM). While the Nano-R can be operated with little or no understanding of the components of an AFM, Pacific Nanotechnology recommends that those who are new to AFMs first take the time to read the AFM Tutorial on page xxi. Some understanding of the components and theory of an AFM system will greatly facilitate your ability to get optimal results from the Nano-R™ AFM.

AFM HISTORY

When we think of microscopes, we typically 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.

The atomic force microscope (AFM), developed in the mid 1980's, uses a sharp probe to magnify surface features. With an AFM, it is possible to image an object's surface topography with extremely high magnifications, up to 1,000,000x. Furthermore, the magnification of an AFM is made in three dimensions: the horizontal X-Y plane and the vertical Z dimension. As acknowledged by Bennig and Roher[9], the inventors of the tunneling microscope, such a powerful technique has its origins in the stylus profiler.

9. G. Bennig and H. Rohrer, Scanning Tunneling Microscopy-From Birth to Adolescence, Rev. of Mod. Phys, Vol 59, No. 3, 1987, P 615

STYLUS PROFILERS

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. Figure a illustrates an example of an early profiler. This profiler, invented by Schmalz[10] 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.

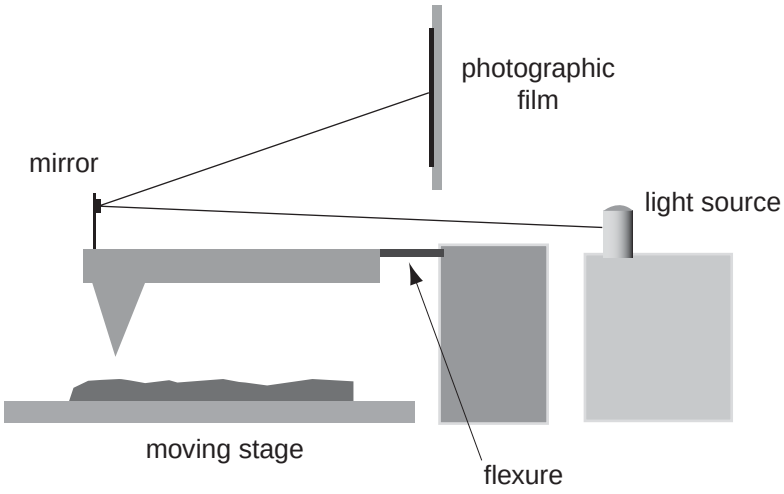

Figure a. Example of a surface profiler made in 1929.

A common problem with stylus profilers was the possible bending of the probe due to collisions with surface features. Such "probe bending" was a result of horizontal forces on the probe caused when it encountered large features on the surface. This problem was first addressed by Becker[11] in 1950 and later by Lee[12]. Both Becker and Lee suggested oscillating the probe from a null position above the surface to make contact with the surface. Becker remarked that the detail of images measured using this vibrating profile method would depend on the sharpness of the probe.

10. Uber Glatte und Ebenheit als physikalisches und physiologishes Problem, Gustev Shmalz, Zeitchrift des Vereimes deutscher Ingenieurte, Oct 12, 1929, pp. 1461-1467
11. U.S. Patent 2,7288,222
12. UK Patent 2,009,409

In 1971, Russell Young[13] demonstrated a non-contact type of stylus profiler. In his profiler, called the Topographiner, Young used the fact that, for electrically conductive samples, the electron field emission current between a sharp metal probe and a surface is very dependent on the probe-sample distance. In the Topographiner, 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 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.

SCANNING TUNNELING MICROSCOPES AND ATOMIC FORCE MICROSCOPES

In 1981, researchers at IBM were able to utilize the methods first demonstrated by Young to create the scanning tunneling microscope[14] (STM). Bennig and Rohrer 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: Bennig and Rohrer 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 only worked on electrically conductive samples.

A major advancement in profilers occurred in 1986 when Bennig and Quate[15] demonstrated the AFM. Using an ultra-small probe tip at the end of a cantilever, the AFM 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, similar to the technique first used by Schmalz, could be used for measuring the motion of the cantilever. In their paper, Bennig and Quate proposed that the AFM could be improved by vibrating the cantilever above the surface.

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13. R. Young, J. Ward, F. Scire, The Topografiner: An Instrument for Measuring Surface Microtopography, Rev. Sci. Inst., Vol 43, No 7, p 999
14. G. Bennig, H. Rohrer, Ch. Gerber, E. Weibel, Surface Studies by Scanning Tunneling Microscopy, Vol. 49, No 1, 1982, p 57
15. G. Bennig, C.F. Quate, Ch. Geber, Atomic Force Microscope, Phys. Rev. Letters, Vol. 56, No 9, p 930

The first practical demonstration of the vibrating cantilever technique in an AFM was made in 1987 by Wickramsinghe[16], using an optical interferometer to measure the amplitude of a cantilever's vibration.

Oscillation amplitudes of between 0.3 nm and 100 nm were achieved with this optical technique. Because the probe came into close contact with the surface upon each oscillation, Wickramsinghe was able to sense the surface materials; the differences between photo-resist and silicon were readily observed.

16. Y. Martin, C.C. Williams, H.K. Wickramasinghe, Atomic Force Microscope-Force Mapping and Profiling on a sub 100-Å scale. J. Appl. Phys. Vol 61, No 9, 1987, p 4723

NANOSCIENCE & NANOTECHNOLOGY OVERVIEW

Approximately 15 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 now 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 are able to 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 can routinely study the spectra of atoms and molecules. Several decades ago, biochemists began to discover the usefulness of particular types of molecules such as proteins, enzymes, and DNA.

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 for a large number of molecules.

SO WHAT'S NEW?

All the revolutions in science and technology are facilitated by many driving forces occurring simultaneously. The nanotechnology revolution, too, is being driven by a number of developments, ideas, and technical advancements, the primary ones being:

Instruments that measure & manipulate atoms and molecules

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

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

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 time and the size of electronic devices such as transistors. 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, therefore, to discover new methodologies for creating electronic devices of this size and smaller.

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 and transmit information on smaller spaces and transmit information with faster methodologies.

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

Researchers in the life sciences have 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 dimensions on the order of a few hundredths of an inch. An artist could paint pictures; a potter could make dishes and pots. With the industrial revolution, it became possible to routinely make machines with tolerances of a few thousandths of an inch (25 to 100 microns), which gave way to the invention of the steam engine, railroads, the car, and the airplane.

With MEMS technology, it is now possible to use machining technologies to create machines smaller than the width of a human hair. This ability is presently used in the sensors that activate airbags 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 we collect from the macroscopic world in which we live. Presently, biologists, chemists, physicists, and engineers have only a mental picture of what is occurring on 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 in a box 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 revolution, 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. For example, the smallest transistors we make in a factory today are about 130 nanometers wide. With future nanotechnology advancements, engineers will be able to make chips that have transistors 2-3 nanometers wide. Today, cosmetic manufacturers use liposomes with diameters of a few tens of nanometers to reduce the dehydration of skin.

We expect that 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 discussed as part of the nanotechnology revolution are fully implemented, what is possible? At this point, many of the possibilities being discussed seem like science fiction.

We can only imagine what is possible…

Imagine:

- All of recorded history will fit in a package small enough to carry in our pockets. This includes all written documents, music, and movies.
- Our world will be safer because the computers and sensing systems that fit in a package the size of a pill will be able to warn us of dangers.
- Life will be extended because we can create systems and modules that replicate the functions and systems in our bodies.
- New types of “quantum computers” will make calculations billions of times faster than today's digital computers.
- 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 AFM'S CONTRIBUTION TO NANOTECHNOLOGY?

Measurement

An atomic force microscope (AFM) creates a highly magnified, three-dimensional image of a surface. The image is generated by monitoring the motion of an atomically sharp probe as it is scanned across a surface. With an AFM, scientists and engineers can directly view and measure surface features having dimensions on the order of a few nanometers, including single atoms and molecules.

An AFM makes it possible to measure more than the physical dimensions of a surface, as there is a "physical" interaction of the probe with the surface. For example, by lightly pushing against the surface with the probe, it is possible to measure surface hardness. Also, the degree of ease with which the probe glides across the surface is a measure of the surface "friction."

Modification

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

Manipulation

An AFM probe can be used 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 for generating the motions of the probe. Some interfaces measure the locations of particles, such as microspheres on a surface, and then automatically move the spheres to a pre-established location. In another type of interface, called the nanomanipulator, the motion of the probe follows the motion of the user's hand. When you move your hand up and down, the probe moves up and down. This kind of interface also allows the user to "feel" and "touch" a surface.

AFM Tutorial

INTRODUCTION

This section serves as an introduction to how an AFM works. With a basic understanding of the technologies employed in an AFM and how they are implemented in the design and operation of the instrument, you can obtain optimal results from your Nano-R AFM.

CONCEPTS & TECHNOLOGIES

DIMENSIONS AND MAGNIFICATION

An AFM is optimized for measuring surface features that are extremely small, therefore it is important to be familiar with the dimensions of the features being measured. An AFM is capable of imaging features as small as a carbon atom (~ 0.25 nanometers in diameter) and as large as the cross section of a human hair (~ 80 microns in diameter).

The common unit of dimension used for making measurements in an AFM is the nanometer (nm), one billionth of a meter:

1 meter = 1,000,000,000 nanometers

1 micron (µm) = 1,000 nanometers

Another common unit of measure is the Angstrom (Å), a tenth of a nanometer:

1 nanometer = 10 Angstroms

Magnification in an AFM is the ratio of the actual size of a feature to the size of the feature when viewed on a computer screen. Thus, when an entire cross section of a human hair is viewed on a 500 mm (20-inch) computer monitor, the magnification can be expressed as:

Magnification = 500 mm / .08 mm = 6,250x

In the case of extremely high resolution imaging, the entire field of view of the image may be 100 nm. In this case, the magnification on a 500 mm computer screen is:

Magnification = 500 mm / (100 nm*1 mm / 1,000,000 nm) = 5,000,000x

PIEZOELECTRIC CERAMIC TRANSDUCER

Precise mechanical motion in an AFM is created from electrical energy using an electromechanical transducer. The electrical motor used in a washing machine is the most common example of an electromechanical transducer. The electromechanical transducer most commonly used in an AFM is the piezoelectric ceramic.

A piezoelectric material undergoes a change in geometry when it is placed in an electric field. The amount and direction of motion depends on the type of piezoelectric material, the shape of the material, and the field strength. Figure b shows the motion of a piezoelectric disk when exposed to an electric potential.

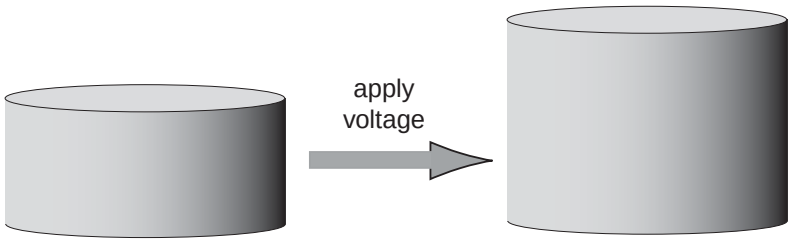


Figure b. When a voltage is applied to the top and bottom surface of the piezoelectric disc, the disc expands.

A typical piezoelectric material will expand by about 1 nm per applied volt. Therefore, larger motions can be attained by making piezoelectric transducers with hundreds of layers of piezoelectric materials, as illustrated in Figure c.

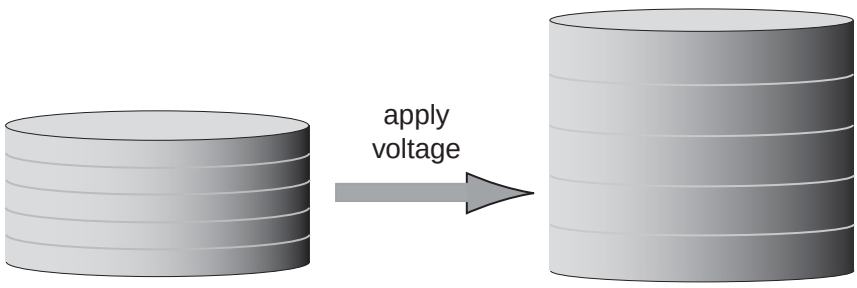


Figure c. When a voltage is applied to the top and bottom surface of a stack of piezoelectric disks, the entire stack expands.

The amount of expansion of the whole stack depends on the applied voltage, the piezo material, and the number of disks. By using one thousand layers of piezo-electric material, it is possible to get motions as large as 1000 nm per volt, or 0.1 mm of motion with 100 volts.

FORCE SENSORS

The construction of an AFM requires a force sensor to measure the forces between a small probe and the surface being imaged. A common type of force sensor utilizes the relationship between the motion of a cantilever and the applied force. The relationship is given by Hook's law:

$$F = - K * D$$

where:

- K is a constant which depends on the material and dimensions of the cantilever
- D is the motion of the cantilever.

For a cantilever made of silicon that has dimensions of:

$$L = 100\ \mu m,\ W = 20\ \mu m,\ T = 1\ \mu m,$$

the force constant, K, is approximately 1 newton / meter. Therefore, a force of 1 nanonewton is required to move the cantilever 1 nm.

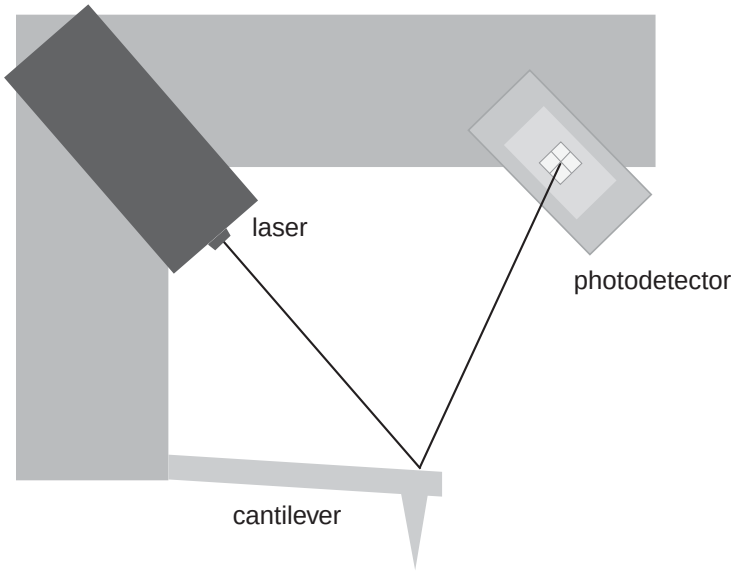


Figure d. The light lever method for sensing the motion of the cantilever.

The motion of the cantilever can be measured with the “light lever” method, as illustrated in Figure d. A laser beam is reflected off the back side of the cantilever and onto a photo-detector. Deflection of the cantilever causes the laser beam to move across the surface of the photo-detector.

The motion of the cantilever is then directly proportional to the output of the photo-detector. Motions as small as 1 nm are routinely measured by AFMs using this method.

FEEDBACK CONTROL

Feedback control is commonly used for keeping the motion of one object in a fixed relationship to another object. A simple example of feedback control occurs when you walk down a sidewalk. As you walk, you constantly control your motion by viewing the edge of the sidewalk. If you begin to walk off the sidewalk, you correct your motion so that you stay on the sidewalk. Feedback control is used for many everyday applications, including the automatic controls in airplanes and the thermostat controls in buildings. In an AFM, feedback control is used to keep the probe in a “fixed” relationship with the surface while a scan is measured.

AFM THEORY & INSTRUMENTATION

The theory and operation of an AFM is similar to that of a stylus profiler. The primary difference is that probe forces on the surface are much smaller in the AFM. Because of this, smaller probes can be used, and a much higher resolution can be achieved.

In an AFM, a constant force is maintained between the probe and sample while the probe is raster scanned across the surface. By monitoring the Z motion of the probe as it is scanned, a three dimensional image of the surface is constructed.

The constant force is maintained by measuring the force on the cantilever with the light lever sensor and by using a feedback control electronic circuit to control the position of the Z piezoelectric ceramic. The motion of the probe over the surface is generated by piezoelectric ceramics that move the probe and force sensor across the surface in the X and Y directions. See Figure e.

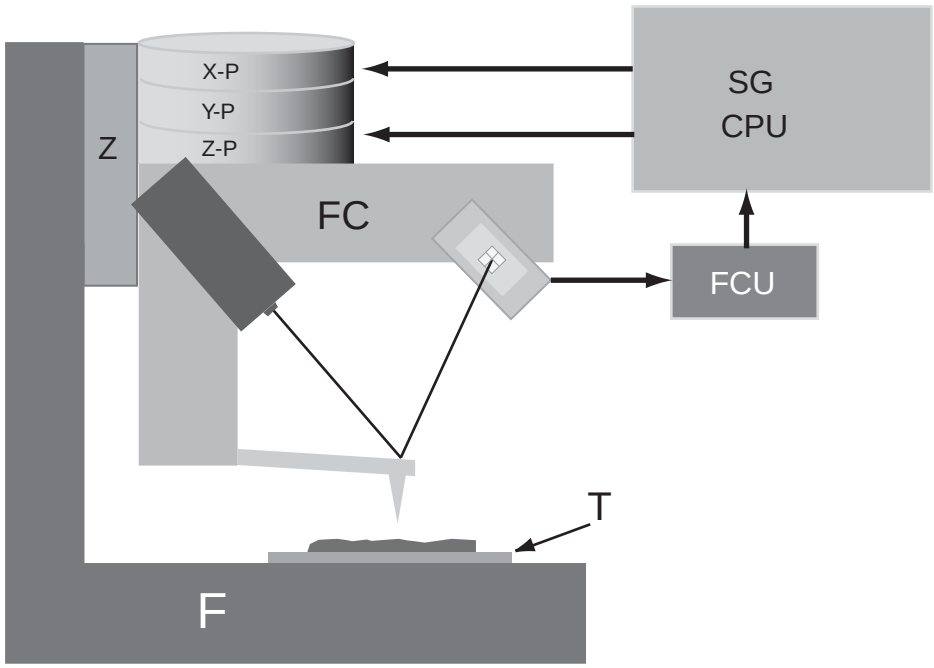


Figure e. Main components and subsystems of an AFM system.

Z - coarse Z motion translator: Moves the AFM head towards the surface so that the force sensor can measure the force between the probe and sample. The motion of the translator is usually about 10 mm.

T - coarse X-Y translation stage: Positions the section of the sample to be imaged directly under the probe.

X-P & Y-P - X and Y piezoelectric transducers: Move the probe over the surface in a raster motion when an image is measured.

FS - Force Sensor: Measures the force between the probe and the sample by monitoring the deflection of the cantilever.

Z-P - Z piezoelectric ceramic: Moves the force sensor and probe in the vertical direction in response to the measured deflection of the cantilever as the probe is scanned across the surface.

FCU - Feedback control unit: Takes in the signal from the light lever force sensor and outputs the voltage that drives the Z piezoelectric ceramic. This voltage refers to the voltage required to maintain a constant deflection of the cantilever while scanning.

SG - X-Y signal generator: Controls the raster motion of the probe in the X-Y plane when an image is measured.

CPU - Computer: Used for setting the scanning parameters (such as scan size, scan speed, and feedback control response) and for visualizing images captured with the microscope.

F - Frame: A solid frame supports the entire AFM instrument. The frame must be very rigid in order to prevent vibrations between the tip and the surface.

NOTE: Not shown in Figure e is an optical microscope, which is essential for locating features on the sample surface and for monitoring the probe approach process.

TAKING IMAGES

Taking an image of a sample with an AFM involves the following basic steps:

1. Install a probe in the microscope, and align the light lever sensing system.
2. Position the region of interest on the sample directly under the AFM probe, using the X-Y translation stage and the optical microscope.
3. Engage the Z translation stage to bring the probe to the surface.
4. Start the scanning of the probe over the surface, and monitor the resulting AFM image on the computer screen.
5. Save the image on a computer disk.

RESOLUTION

Traditional microscopes have only one measure of resolution: the resolution in the plane of an image. An AFM has two measures of resolution: in the X-Y plane of the measurement surface (in-plane resolution) and in the direction perpendicular to the surface (vertical resolution).

In-Plane Resolution: The in-plane resolution depends on the geometry of the probe used for scanning. In general, the sharper the probe, the higher the resolution. The theoretical line scans in Figure f illustrate the difference between using a sharp probe and a dull probe to measure two spherical features on a sample surface. The sharper probe will result in a higher resolution image.

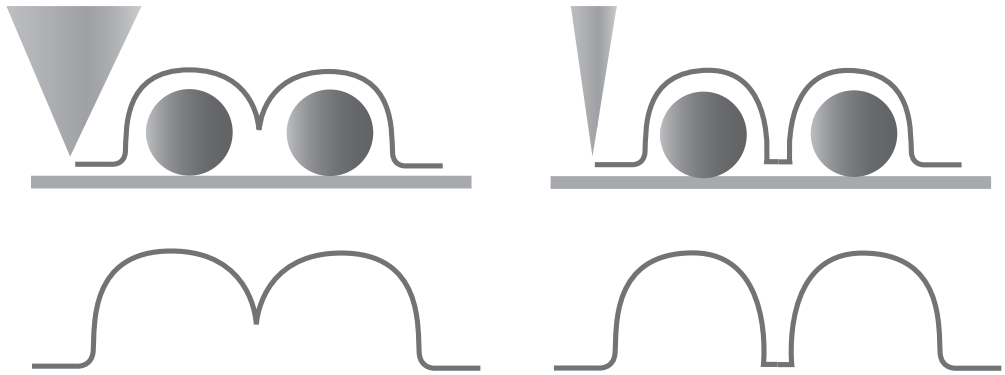


Figure f. Using a dull probe vs. a sharp probe to measure spherical features.

Vertical Resolution: The vertical resolution in an AFM is established by relative vibrations of the probe above the surface. Sources of vibrations include acoustic noise, floor vibrations, and thermal vibrations. Getting the maximum vertical resolution requires minimizing these vibrations.

PROBE SURFACE INTERACTIONS

The strongest forces between the probe and surface are the mechanical forces that occur when the atoms on the probe physically interact with the atoms on a surface. However, other forces between the probe and surface can have an impact on an AFM image. These include surface contamination, electrostatic forces, and surface material properties.

Surface contamination: In ambient air, all surfaces are covered with a very thin layer (< 50 nm) of contamination. This contamination, which can be comprised of water and hydrocarbons, depends on the microscope's operating environment.

When the probe comes into contact with the surface contamination, capillary forces can pull the probe towards the surface.

Electrostatic forces: Insulating surfaces can store charges on their surface, which can interact with charges on the probe or cantilever. Such forces can be so strong that they "bend" the cantilever when scanning a surface.

Surface material properties: Heterogeneous surfaces can have regions of varying hardness and friction. As the probe is scanned across a surface, the probe-surface interaction can change when moving from one region to another. Such changes in forces can give a "contrast" that is useful for differentiating between materials on a heterogeneous surface.

AFM IMAGING MODES

Topography Modes

As the probe at the end of the cantilever is scanned over the sample surface, a constant force between the probe and the sample is maintained. There are two methods for measuring the force on the cantilever as the probe encounters changes in the sample topography. In deflection, or "contact," mode, the deflection of the cantilever is measured directly. In vibrating mode, the cantilever is vibrated, and changes in the vibration properties are measured.

Deflection Mode: Using the feedback control in the AFM, it is possible to scan a sample with a fixed cantilever deflection. Because the deflection of the cantilever is directly proportional to the force on the surface, a constant force is applied to the surface during a scan. While this scanning mode is often called "contact" mode, because the forces of the probe on the surface are often less than a nanonewton, the probe is minimally touching the surface.

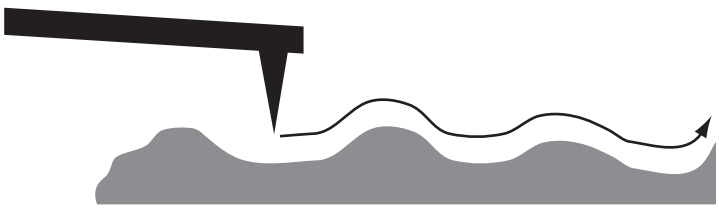


Figure g. Contact mode AFM: the probe directly follows the topography of the surface as it is scanned while a constant force is maintained.

Vibrating Mode: The cantilever in an AFM can be vibrated using a piezoelectric ceramic. When the vibrating cantilever comes close to the sample surface, the amplitude and phase of the vibrating cantilever may change. The feedback unit keeps either the vibration amplitude or phase constant. Changes in the vibration amplitude or phase are easily measured, and the changes can be related to the force on the surface. This technique has many names, including "non-contact" and "intermittent contact" mode. It is important that the tip not "tap" the surface, as this may break the probe or damage the sample.

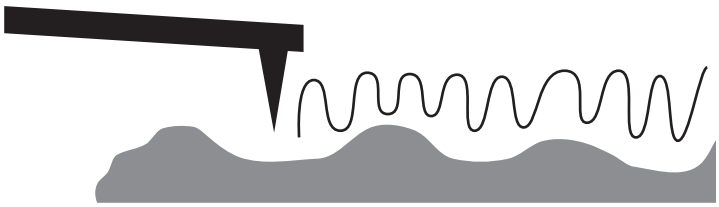


Figure h. In vibrating methods, changes in probes vibrations are monitored to establish the force of the probe onto the surface.

Material Sensing Modes

The interaction of the probe with the surface depends on the chemical and physical properties of the surface. It is therefore possible to measure these interactions and thus "sense" the materials at a sample surface.

Vibrating Material Sensing Mode: The AFM cantilever may be vibrated to measure the force between the probe and sample during a scan. The magnitude of amplitude damping and the amount of phase change of the cantilever depends on the surface chemical composition and the physical properties of the surface. Thus, on a non-homogeneous sample, contrast can be observed between regions of varying mechanical or chemical composition. Typically, in vibrating material sensing mode, if the amplitude is fixed by the feedback unit, then the contrast of the material is observed by measuring phase changes. This technique has many names, including phase mode, phase detection, and force modulated microscopy.

Torsion Modes: In contact mode AFM, it is possible to monitor the torsion motions of the cantilever as it is scanned across the surface. The amount of torsion of the cantilever is affected by changes in topography as well as changes in surface chemical properties. If a surface is perfectly flat but has an interface between two different materials, it is often possible to image the change in material properties. This technique is similar to lateral force microscopy (LFM).

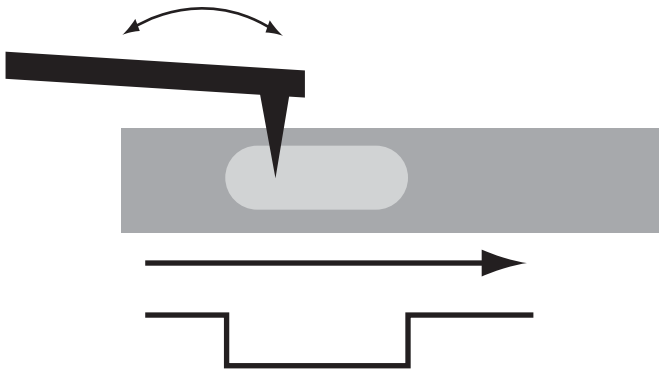


Figure i. Torsion mode: changes in the torsion of the cantilever are an indication of changes in the surface chemical composition.

Chapter 1

Instrument Overview

WARNING: *Before operating the Nano-R AFM, make sure you are familiar with the safety information on page vi.*

CAUTION: To prevent damage to your instrument, probe, and sample, observe all the caution statements in the tutorial chapters (Chapter 2 and Chapter 3).

NANO-R AFM INSTRUMENT SYSTEM

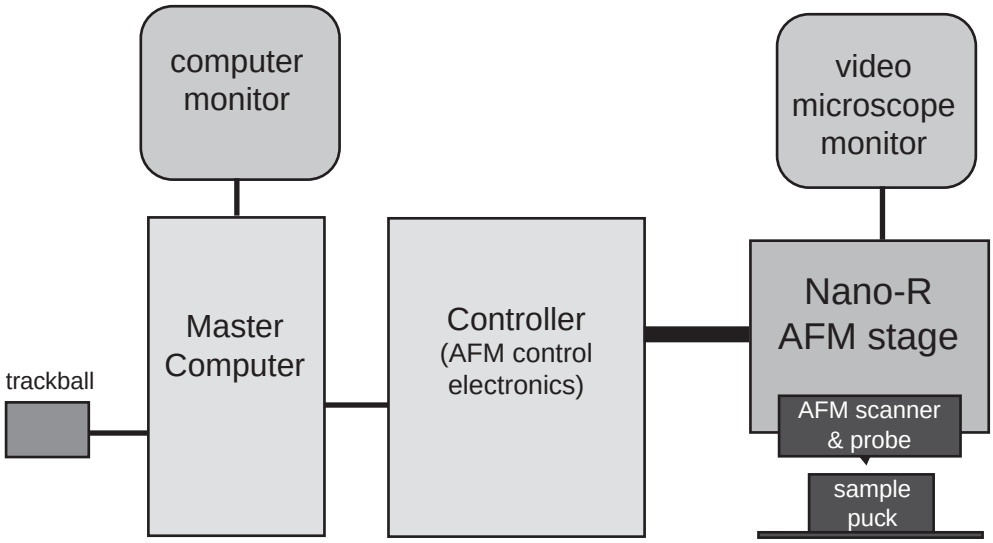


Figure 1.1. Block diagram of Nano-R instrument system.

Nano-R Stage - Includes the AFM scanner, probe, sample puck, video optical microscope, and the AFM scanner's real-time calibration sensors.

Nano-R stage

Master Computer - The IBM PC-type computer is the virtual interface to the Nano-R AFM stage. Pacific Nanotechnology software programs resident on the computer's hard disk are used for measuring, visualization, and analysis of AFM images.

Controller - Contains most of the electronics required for operating the Nano-R stage. It is connected to the Master Computer by a standard Ethernet cable, and to the Nano-R stage by five cables.

Controller

Video Monitor - Displays the optical microscope image of the probe-sample area. In some cases, the computer monitor may be used as the video monitor.

Track ball - Provides an optional way to activate many of the motorized features of the Nano-R stage, including the X-Y stage positioning and the video microscope zoom and focus.

HARDWARE COMPONENTS

NANO-R STAGE

The AFM scanner head rests on three motorized posts, which are used to perform a coarse Z approach of the probe tip to the sample surface. The sample puck rests on a motorized X-Y stage for positioning the sample under the probe. The puck can be easily removed for mounting a sample.

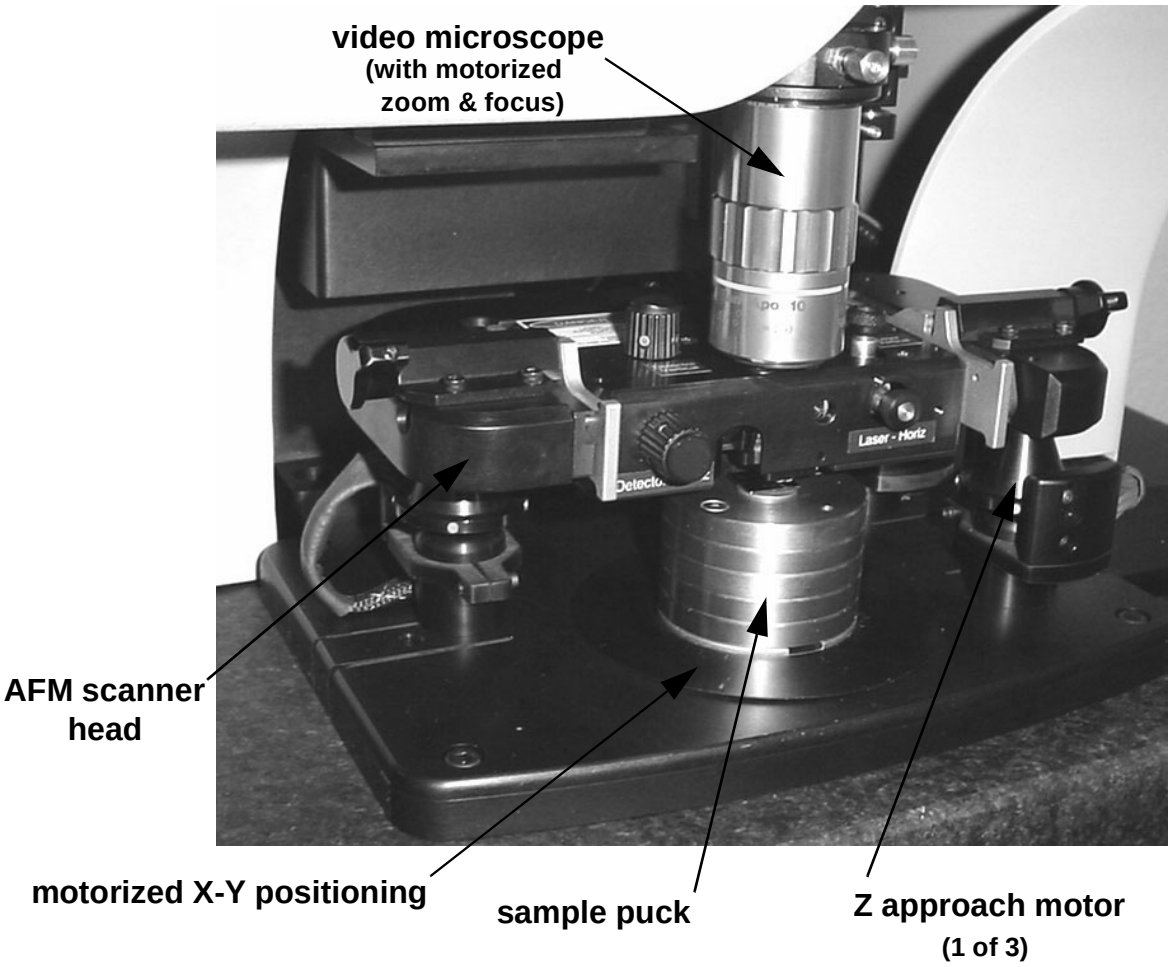


Figure 1.2. Stage components.

AFM SCANNER HEAD

The AFM scanner head contains the components that: 1) measure the force between the probe and the sample, and 2) control the precise positioning of the probe in X, Y, and Z.

The Z piezo component moves the probe vertically in response to changes sensed in the sample surface. The X and Y piezos move the probe over the sample in a raster pattern, which defines the scan region.

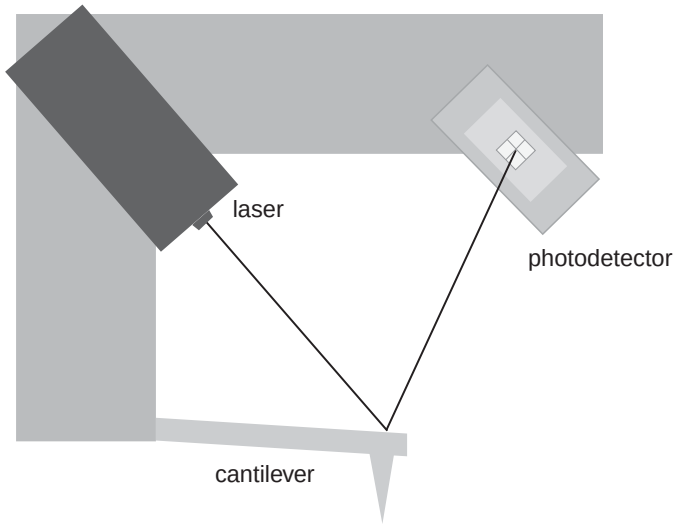


Figure 1.3. Light lever sensing system.

The Nano-R AFM scanner uses a light lever design. A red laser is focused on the back of the cantilever and then projected onto a quad photodiode (photodetector). Two pairs of manual adjustment knobs on the scanner head are used to align the sensing system. One pair controls the position of the laser light on the backside of the cantilever; the other pair moves the photodetector into the light path.

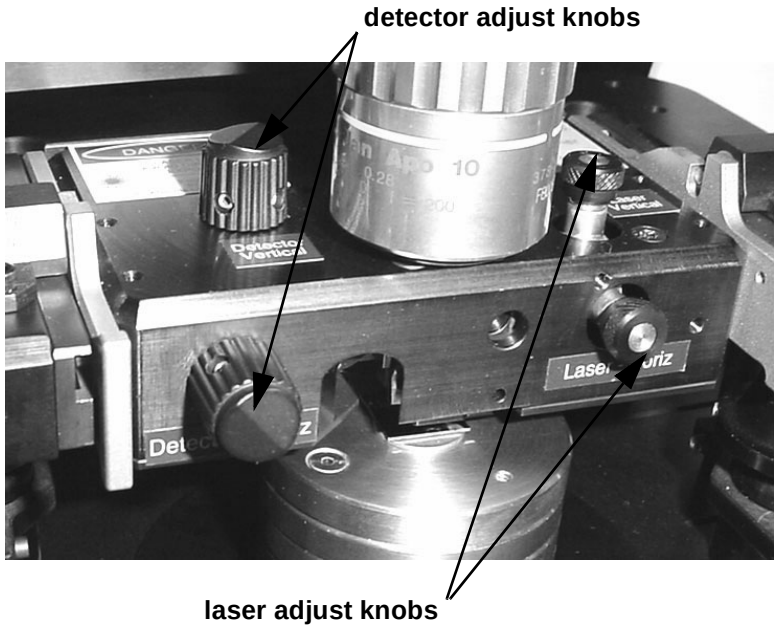


Figure 1.4. Adjustment knobs for laser and detector.

AFM PROBES

CAUTION: Use care when handling AFM probes, as they can break very easily. Always handle with tweezers, and never touch the cantilever.

The Nano-R AFM is shipped with probes for the two basic imaging modes: contact and close contact (vibrating cantilever). The probes come in two marked boxes, 10 probes to a box.

AFM probes

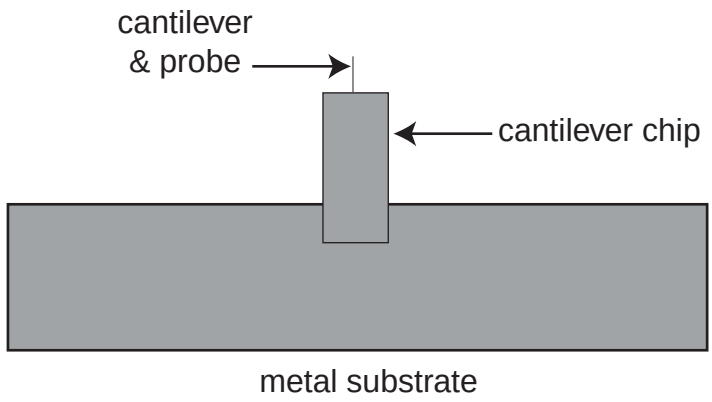


Figure 1.5. PNI AFM probe (top view).

The probe tip extends from the end of a cantilever which is mounted to a chip. The metal substrate that holds the cantilever chip is mounted in the AFM scanner; it is magnetically coupled to the bottom of the scanner.

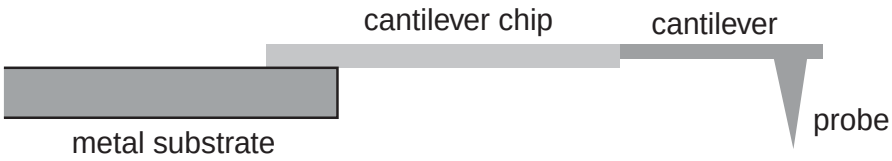


Figure 1.6. PNI AFM probe (side view—not to scale).

The two types of probes appear identical to the naked eye, but under the instrument's optical microscope, you can see that contact cantilevers are significantly longer than close contact cantilevers.

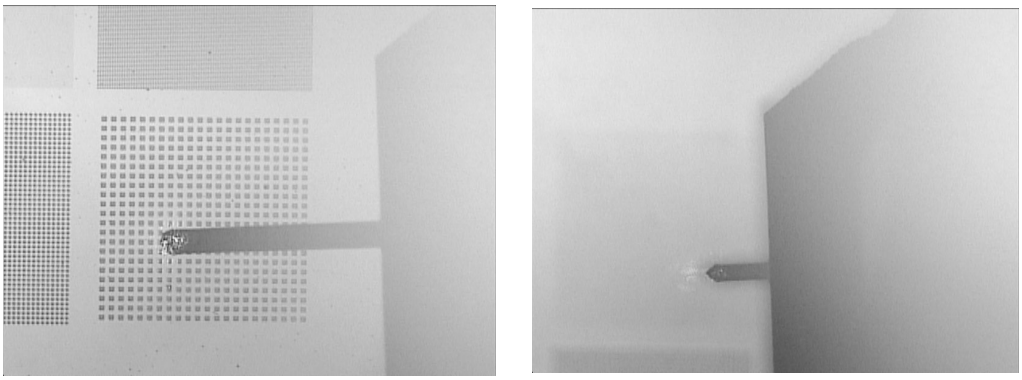


Figure 1.7. The two cantilever types, as seen on the video microscope monitor.

SAMPLE PUCK

The sample to be imaged is mounted on the sample puck. The puck is composed of removable layers so the height of the puck can be adjusted to accommodate different sample sizes (see page 85 for details). The protruding piece on the bottom of the puck fits into the groove on the X-Y stage so it can be safely and easily guided into position under the probe.

Figure 1.8. Sample puck.

PNI REFERENCE

The Nano-R system is supplied with the PNI AFM reference, which is helpful for establishing the performance of your instrument's AFM scanners as well as the optical microscope. The reference also serves as a useful test sample when learning how to use your instrument (the tutorials in this manual are based on this sample).

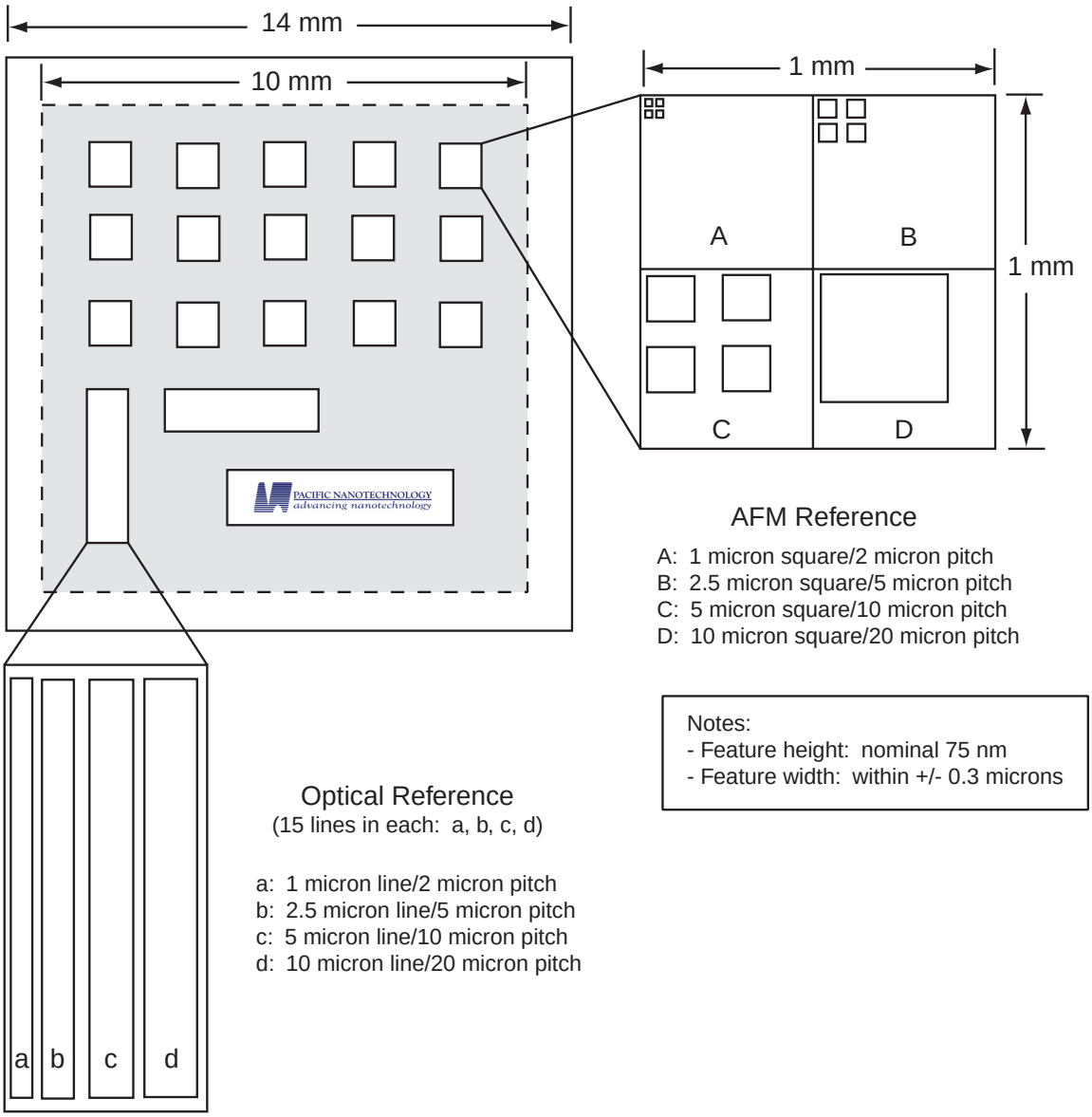


Figure 1.9. PNI reference.

The patterns in the reference are made in a silicon nitride film deposited on a silicon substrate. This combination gives optimal color contrast when viewed with an optical microscope.

The pattern for AFM measurements is composed of four blocks of square features. The features in each block have uniform size and pitch, with each block containing features of a different size, as illustrated in Figure 1.9. This pattern is repeated at 15 locations on the reference. The pattern for optical microscope reference is composed of a series of four sets of parallel lines and a second series perpendicular to the first.

SOFTWARE MODULES

The SPM Cockpit software modules serve three functions:

- acquire AFM data
- process and analyze the acquired data
- display AFM images (contained in the analysis modules)

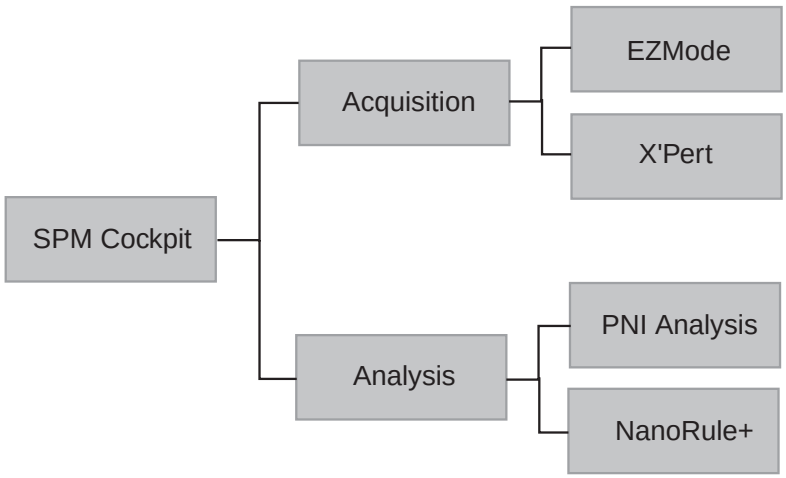
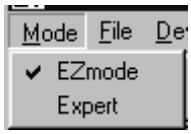


Figure 1.10. SPM Cockpit software modules.

The interfaces for the image acquisition and analysis modules feature tool bars that provide convenient access to the most commonly-used software functions for the given mode of operation. However, regardless of the module (acquisition or analysis) or mode (EZ or X'Pert) you are in, all of the SPM Cockpit software functions are always accessible via the menu items.

Note that the PNI Analysis software is included with all Nano-R AFM systems, and NanoRule+, a more full-featured analysis software package, is available as an option.

ACQUISITION



When you launch the SPM Cockpit software, the acquisition module opens by default. You will be in either EZMode or X'Pert Mode, depending on the mode used in the last session. Use the Mode menu to switch between the two.

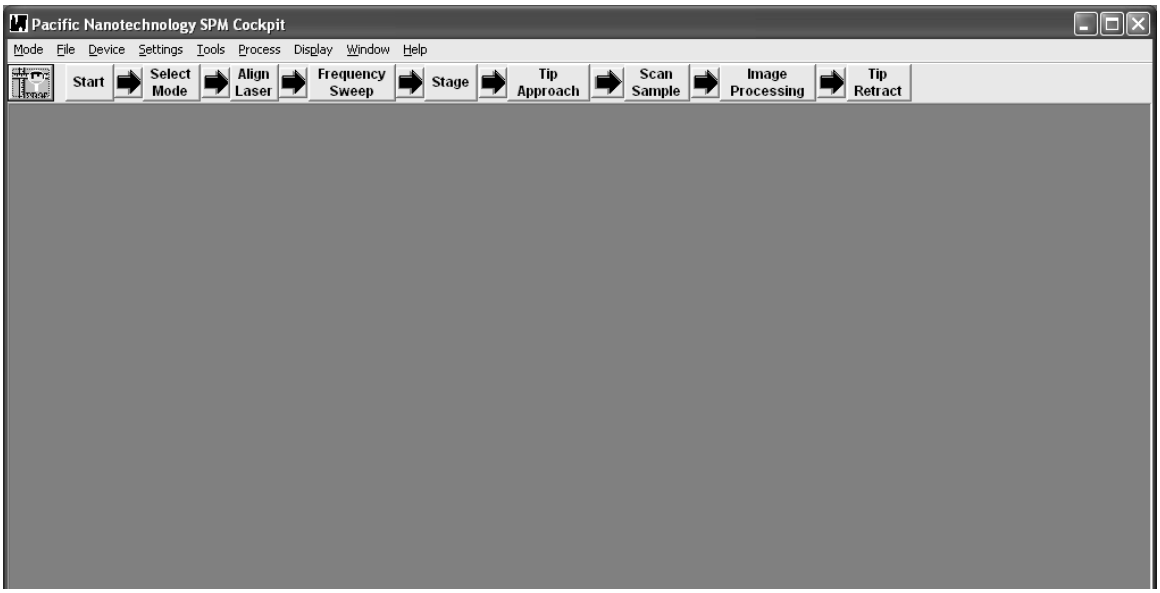


Figure 1.11. Acquisition module main screen, in EZMode.

EZMode is intended for new and occasional AFM users. A set of short-cut buttons forms an easy-to-follow flow chart that takes you through the basic steps for taking an AFM image. Each button opens a dialog offering the choices necessary for accomplishing that step.

Figure 1.12. EZMode short cut buttons.

X'Pert Mode is oriented toward advanced AFM users who want to take advantage of a wider range of choices and features for acquiring an image. The X'Pert Mode short-cut buttons access the functions for accomplishing the same required steps in EZMode, as well as other functions, but the buttons are not necessarily organized into sequential steps.

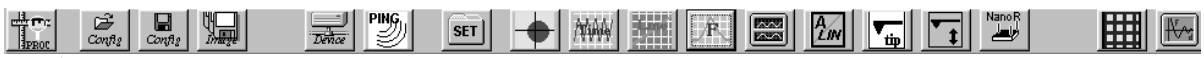


Figure 1.13. X'Pert mode short cut buttons.

ANALYSIS

From the acquisition module, you can switch to the PNI Analysis module by clicking  . A series of short-cut buttons is displayed for easy access to the most commonly used image processing and analysis tools.

Figure 1.14. Analysis module short cut buttons.

To return to the acquisition module, click  .

BASIC IMAGING PROCEDURE

Acquiring an image with the Nano-R AFM requires the following basic steps, whether you are a new, occasional, or advanced user:

1. Launch the SPM Cockpit software
2. Open a configuration file (contact or close-contact).
3. Retract the tip and raise the AFM scanner to provide safe clearance between the probe tip and the sample puck.
4. Load a sample on the sample puck.
5. Install a probe on the AFM scanner.
6. Align the detector.
7. For close contact mode only, set the resonance frequency for the installed cantilever.
8. Locate features for imaging.
9. Bring the probe into contact with the sample.
10. Scan the sample.
11. Perform image processing and analysis routines.
12. Retract the probe from the sample.

Chapter 2

Tutorial: Contact EZMode

BEFORE YOU BEGIN

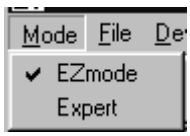
This tutorial follows the steps for an taking a contact AFM image of the PNI AFM reference in EZMode.

WARNING: *Before operating the Nano-R AFM, make sure you are familiar with the safety information on page vi.*

POWERING UP THE SYSTEM

1. Turn on the Master Computer.
2. Launch the SPM Cockpit software.
3. Turn on the Controller.
4. Turn on the video monitor.

SOFTWARE SETUP



1. Select Mode→EZMode.
2. Click the Start button from the EZMode toolbar.

3. Click Retract Tip, and click OK when complete.

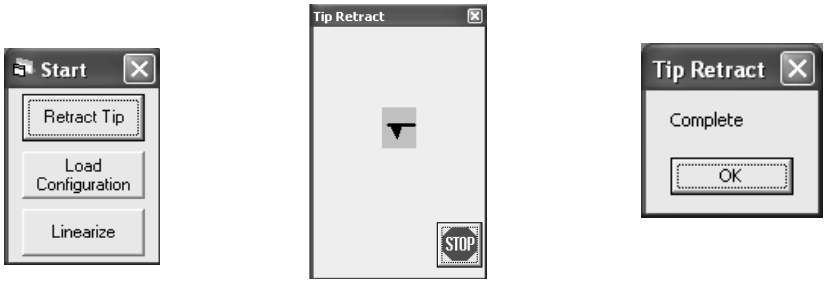


Figure 2.1. Retracting the tip.

4 Click Load Configuration, select the PNI-supplied contact mode configuration file, and click Open.

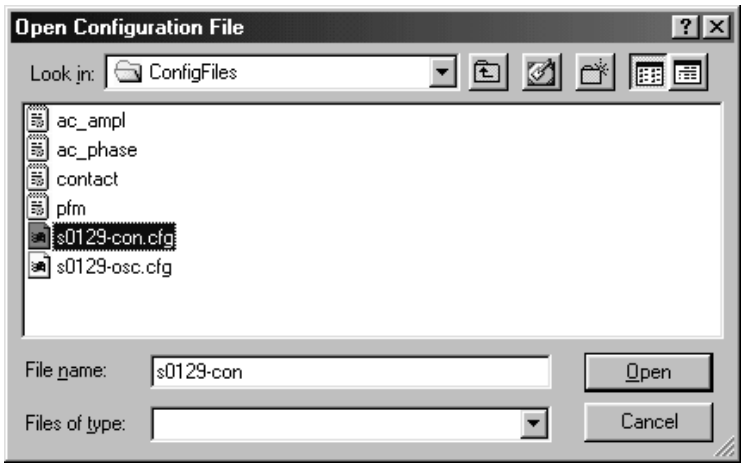


Figure 2.2. Loading a configuration file.

This file should be located in the ConfigFiles folder in the SPM Cockpit directory. It has the format sxxxx-con.cfg, where xxxx is the serial number of your Nano-R system.

5 Click Linearize, check both boxes, and click OK.

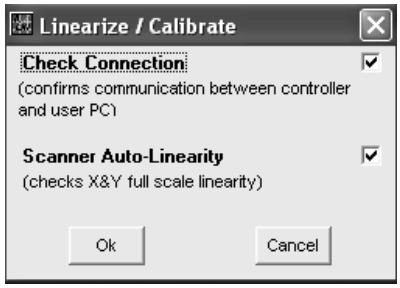


Figure 2.3. Initiate connection confirm and calibration routines.

6 Click OK when the communication between the Master Computer and the Controller is confirmed.

Figure 2.4. Connection confirmed.

If there is no connection, you need to exit the SPM Cockpit software and restart both the Master Computer and the Controller.

7 Click Yes to proceed with the calibration procedure.

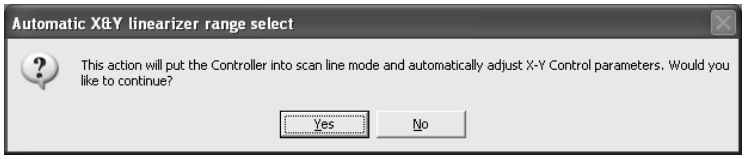


Figure 2.5. Proceed with calibration routine.

8 Click OK when the last step of the calibration process is complete, and then click OK to proceed.

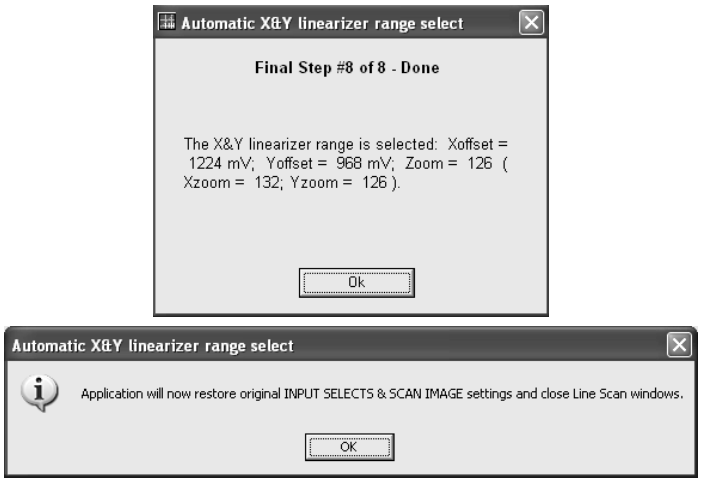


Figure 2.6. Calibration routine complete.

9 Click Select Mode on the toolbar, select Contact in the dialog, and click OK.

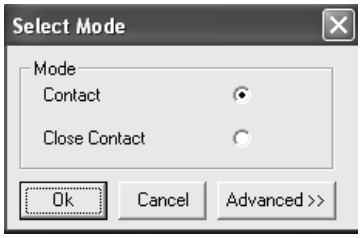


Figure 2.7. Select mode.

10 If the PNI AFM reference is already loaded on the sample puck, skip ahead to page 17 to load a probe, or to page 23 if a contact probe is already loaded.

LOAD A SAMPLE

1 Click Tip Retract from the EZMode toolbar.

2 Click Stage from the EZMode toolbar.

3 Click 

to raise the Z motor until there is at least a few millimeters of clearance between the probe and the sample surface or puck, if no sample is loaded (monitor by eye).

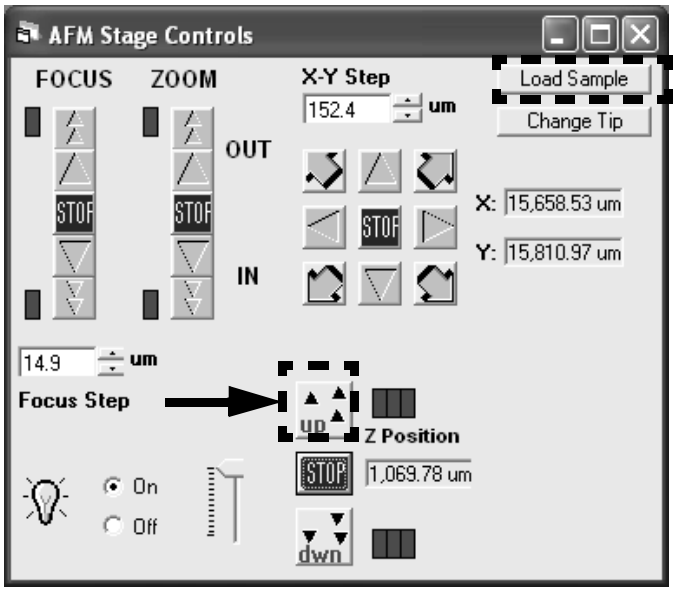


Figure 2.8. Raise the probe tip away from the sample.

CAUTION: To prevent damage to your scanner, probe, and sample, be sure you have retracted the tip and raised the Z scanner (as described in the preceding steps) before moving the puck.

4 Click Load Sample.

The motorized X-Y stage will move the puck towards you, to the limit of its range.

5 Being careful not to touch the probe, slide the sample puck towards you, and then lift it up out of the groove.

Figure 2.9. Remove puck.

6 Use tweezers to mount the PNI AFM reference on the center of the puck.

The sample disk is held in place magnetically.

Figure 2.10. Mounting the sample.

7 Replace the puck on the stage by setting it down so the protruding piece on the bottom fits into the wide part of the groove, and then slide it into position.

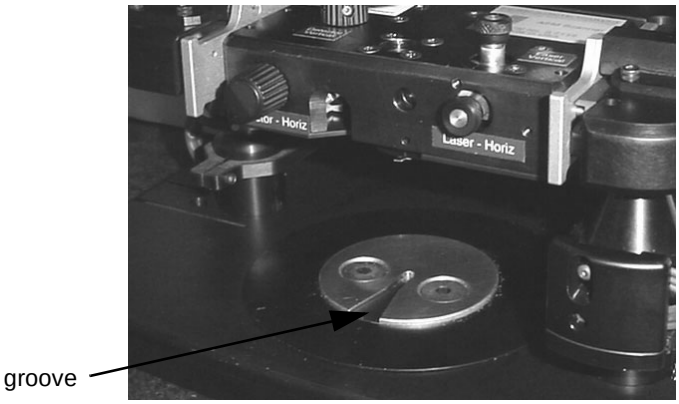


Figure 2.11. Fit the sample puck into the groove on the X-Y stage.

8 Rotate the puck so that the PNI reference sample is square with the scanner head.

9 Select Tools→NanoR Stage to open the AFM Stage Controls window.

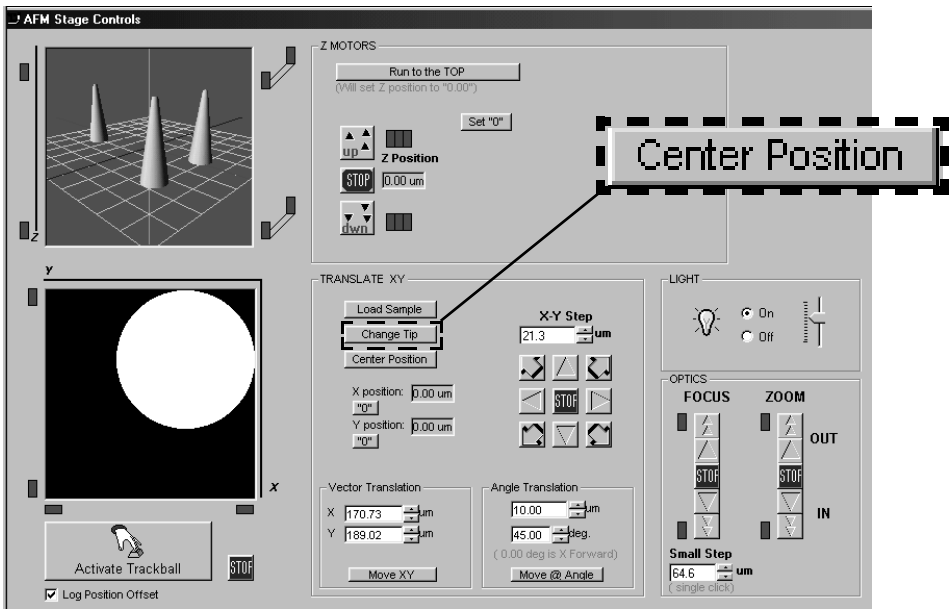


Figure 2.12. Return sample puck to initial X-Y position.

10 Click Center Position.

The motorized X-Y stage will return the puck to its original position

INSTALL A PROBE

To operate in contact mode, you need to use a contact probe. Probes should be stored in the supplied boxes marked "Contact" and "Close-contact," as the difference between the two types of probes is not visible to the naked eye.

1 First, remove the sample puck as described in the section above.

2 Click Stage from the EZMode toolbar, and click Change Tip in the AFM Stage Controls dialog.

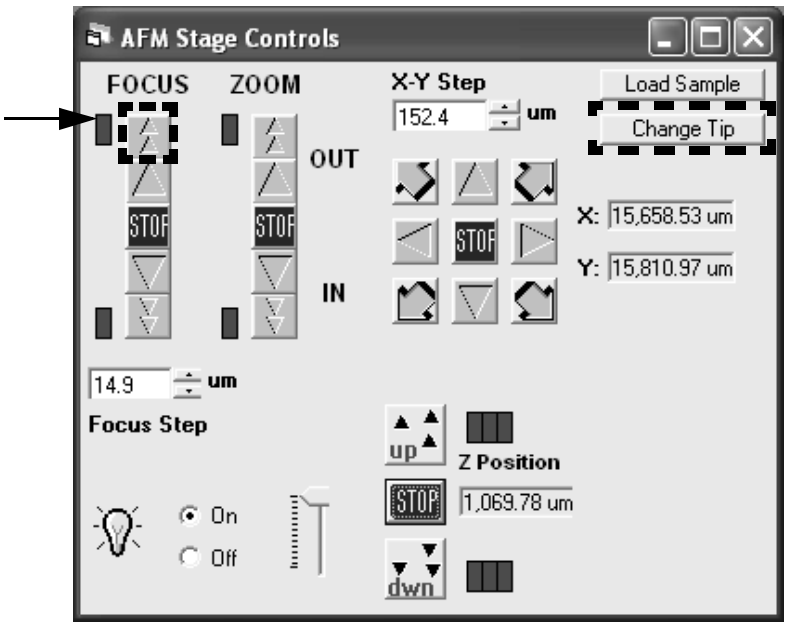


Figure 2.13. Raise the probe tip away from the sample.

The Z motors will raise the scanner to the top of its range.

3 Click the focus button to raise the video objective to the top of its range. The upper indicator will turn from green to red when the objective reaches the top of its range.

4 Click Align Laser from the tool bar.

5 Turn off the laser.

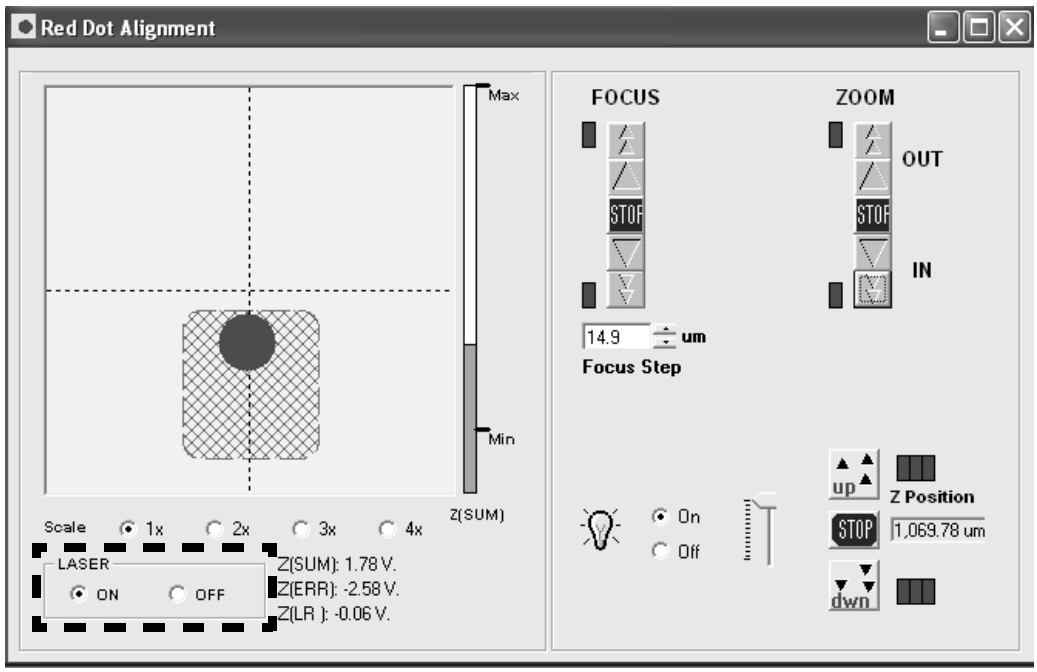


Figure 2.14. Turning off the laser.

WARNING: *To avoid potentially hazardous laser exposure, be sure to turn off the laser before rotating the scanner into the probe exchange position.*

6 Turn the probe exchange knobs on the side of the scanner head down (away from you) 1/4 turn (Figure 2.15).

The scanner head will slide out about an inch.

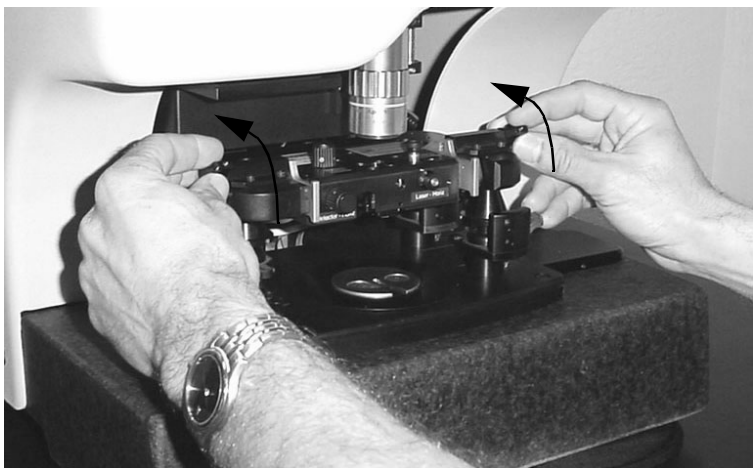


Figure 2.15. Turn the knobs to disengage the scanner head.

7 Grasp the handles on the front of the scanner head (Figure 2.16), and gently slide the scanner head all the way towards you.

Figure 2.16. Slide the scanner head toward you.

8 Carefully rotate the scanner head up about 90 degrees, as shown in Figure 2.17.

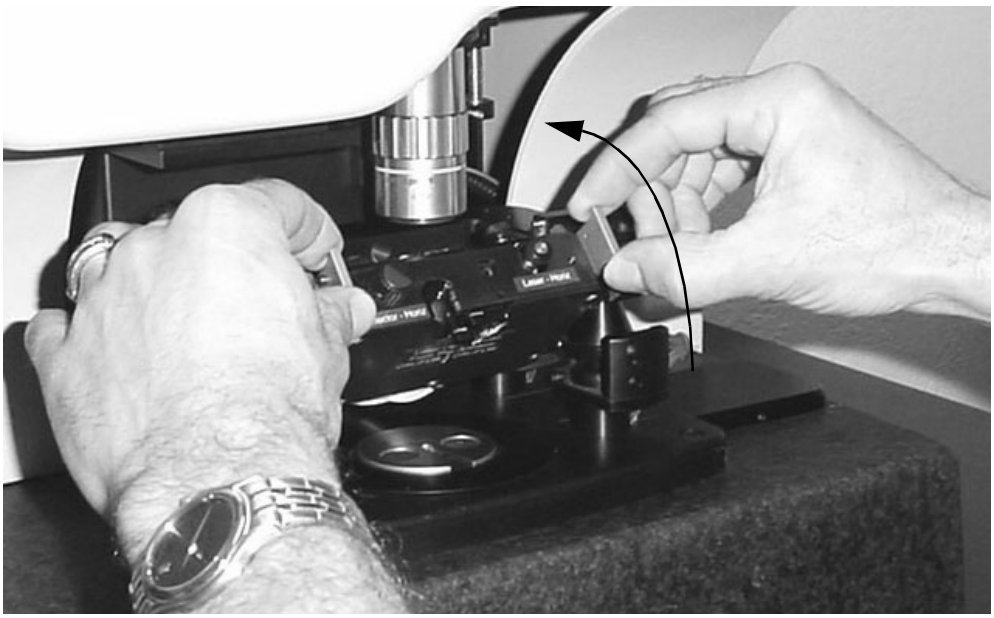


Figure 2.17. Rotate the scanner head.

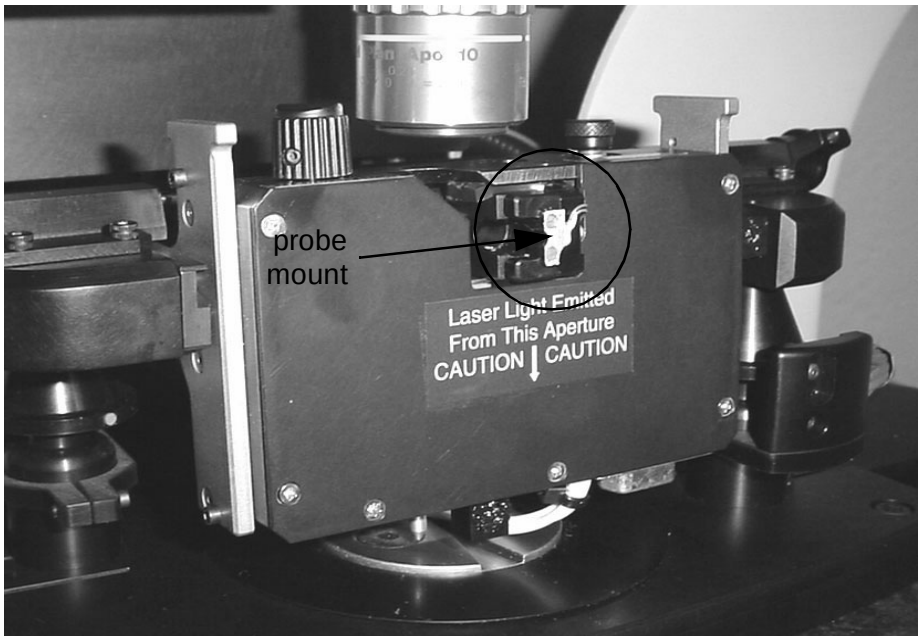


Figure 2.18. Probe exchange position.

CAUTION: Handle AFM probes with care. The cantilever can break off easily if it touches anything or snaps down too forcefully on the magnetic mounting surface on either the scanner or in the probe box.

Probe handling: When loading or removing a probe, pivot the substrate on the edge opposite the cantilever, as shown in Figure 2.19. This will protect the cantilever from striking the magnetic mounting surface, and it will prevent the substrate from snapping down too forcefully, which may damage the probe.

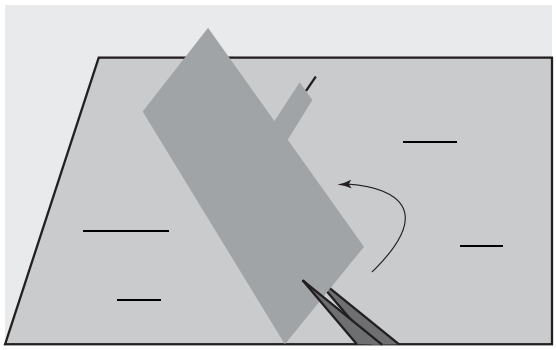


Figure 2.19. Probe handling.

9 To remove a probe:

a) Use tweezers to grasp the metal substrate as indicated in Figure 2.22.

b) Carefully rotate the tweezers so the cantilever side of the substrate lifts up off the magnetic mount first.

c) Set the probe down onto the magnetic strip in the probe box so that the side of the substrate opposite the cantilever makes contact first.

d) Carefully rotate the tweezers so the cantilever side of the substrate comes down onto the magnetic surface as gently as possible.

10 To install a new probe:

a) Use tweezers to nudge a probe so that the substrate extends over the edge of the magnetic strip in the probe box (Figure 2.20).

b) Grasp the metal substrate, and carefully rotate the tweezers so the cantilever side of the substrate lifts up off the magnetic strip first, as shown in Figure 2.21.

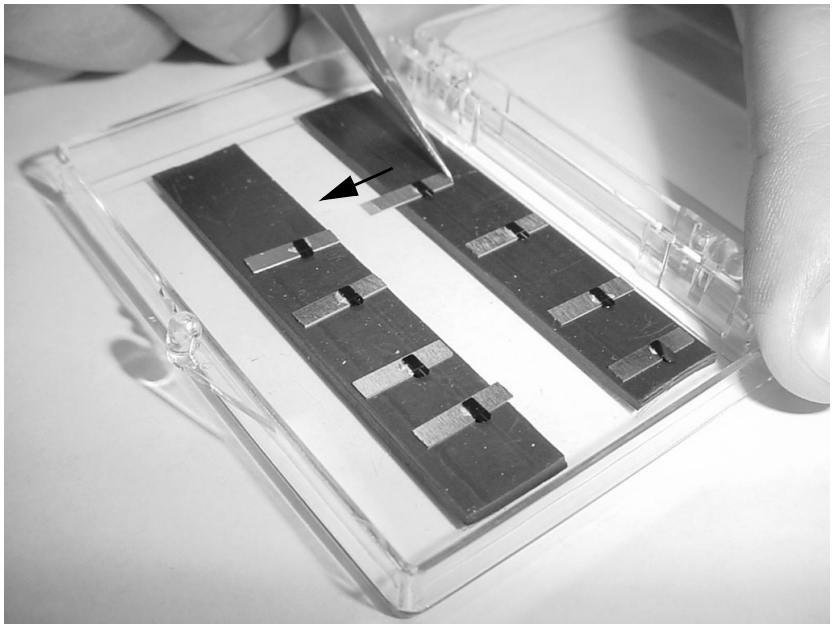


Figure 2.20. Nudge the probe into position.

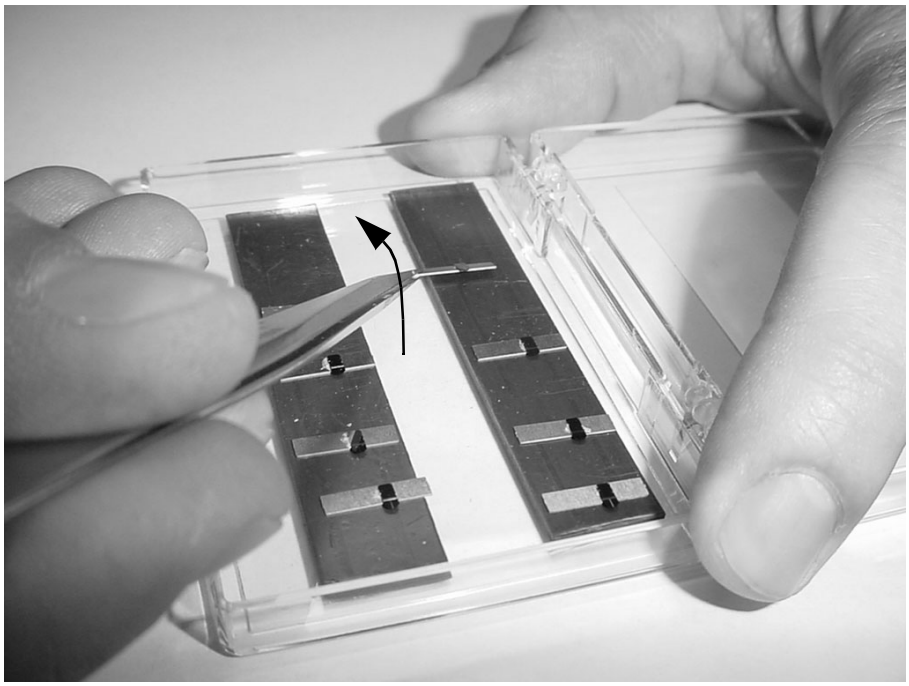


Figure 2.21. Lift the probe, cantilever side first.

c) Place the probe onto the magnetic mount so the side of the substrate opposite the cantilever fits into the “L.”

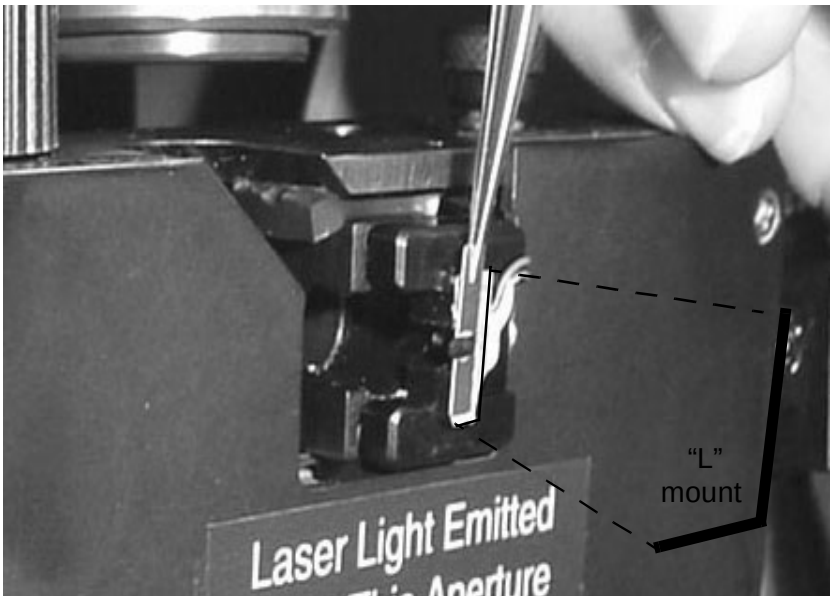


Figure 2.22. Mounting the probe.

d) Use the tweezers to push the substrate flush against the "L," as shown in Figure 2.23.

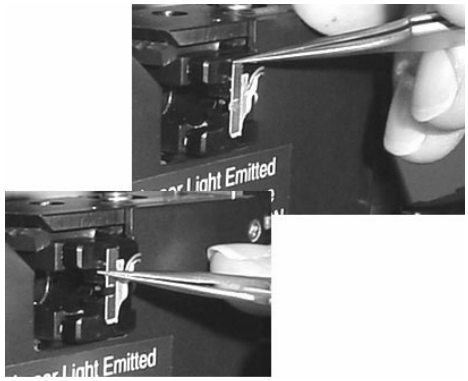


Figure 2.23. Push the probe substrate flush against the "L" mount.

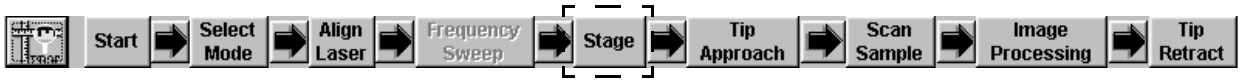
11 Hold the scanner head by the handles, and rotate it back to the level position.

12 Gently slide the scanner back towards the stage until you feel some resistance.

13 Turn the probe exchange knobs up (1/4 turn) to lock the scanner head into place.

Now you can replace the sample puck, as described above (page 16).

ALIGN THE DETECTOR



1 Click Stage from the EZMode toolbar.

2 Use the focus controls to bring the probe tip into focus on the video monitor.

Focus controls: Click  to adjust the focus a single step, or hold it down for continuous motion. Click  to initiate a large, continuous movement of pre-set duration.

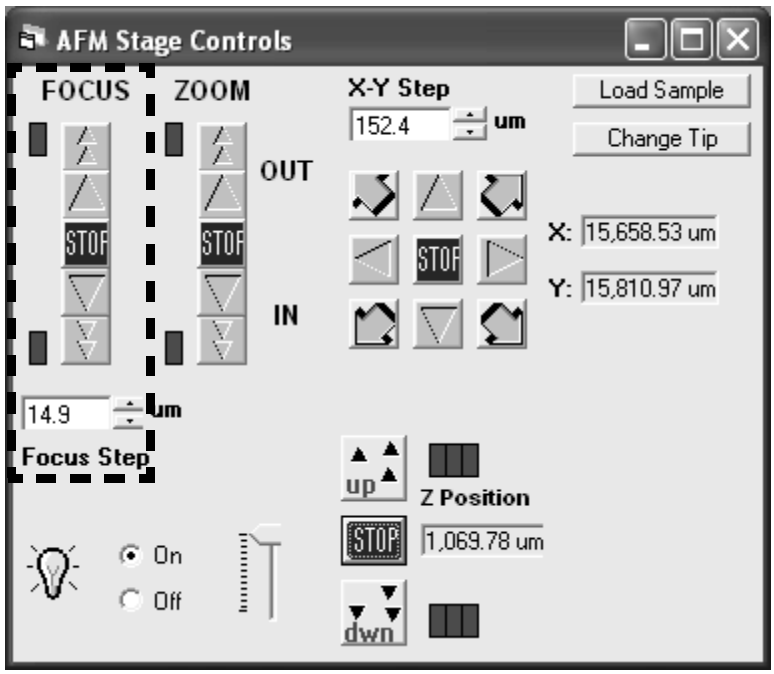
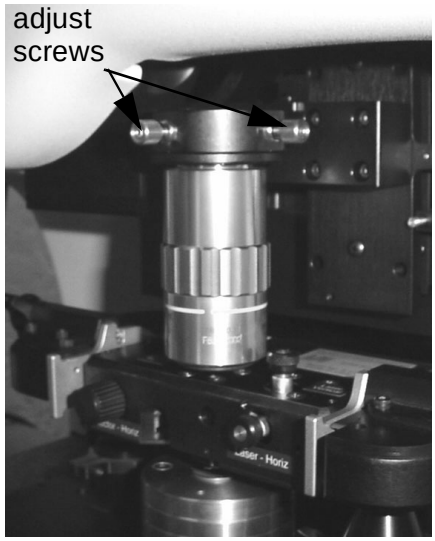


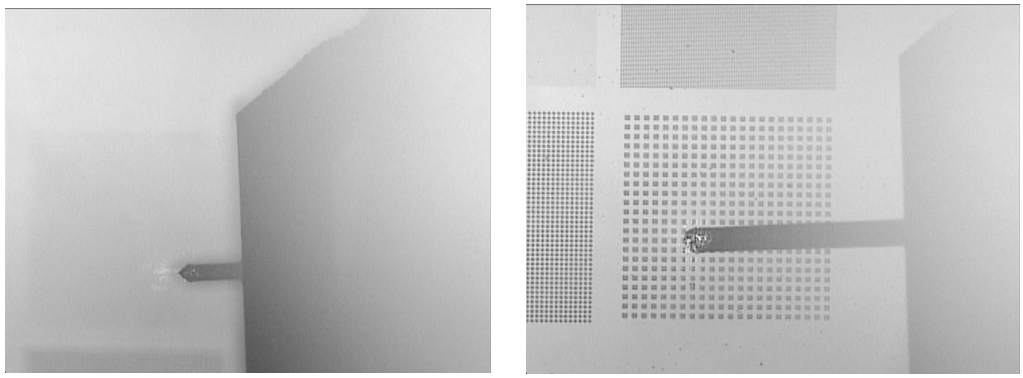
Figure 2.24. Focus controls.

If you cannot find the probe on the monitor:

- The probe may not have been installed properly. Repeat the probe installation procedure to make sure the probe is seated squarely in the "L" mount (page 17).
- The objective's field of view may need to be adjusted in X-Y, using the adjust screws. This is usually necessary when switching between a contact and close-contact probe, due to the difference in size.



You can confirm that you have installed a contact cantilever by noting the difference in length between contact and close-contact cantilevers, as shown in Figure 2.25.



close-contact cantilever contact cantilever

Figure 2.25. Contact vs. close-contact probes.

3 Click **Align Laser** on the toolbar.

4 Turn on the laser.

The red dot alignment procedure has 3 goals:

- position the laser spot on the back of the cantilever
- position the photodetector in the center of the reflected laser beam
- achieve a minimum overall measured signal strength

5 Watch the video monitor as you adjust the laser alignment knobs on the scanner head to bring the laser spot onto the back of the cantilever.

The laser spot should be centered on the cantilever, not too close to the end, as shown in Figure 2.26.

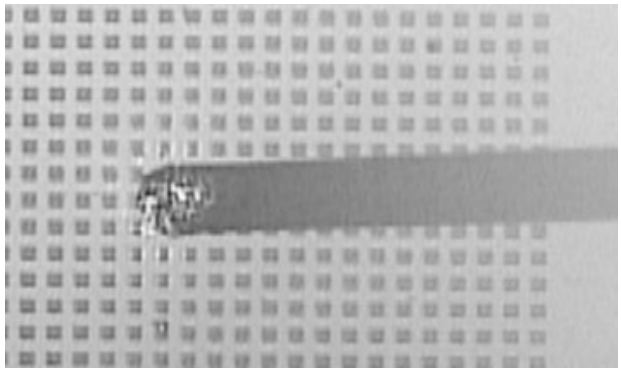


Figure 2.26. Centering the laser spot on the cantilever.

6 Watch the red dot (in the Red Dot Alignment window) as you turn the detector alignment knobs to bring the red dot into the top of the green box.

The red dot should be positioned just below the upper border of the box and be centered along the vertical axis, as shown in Figure 2.27.

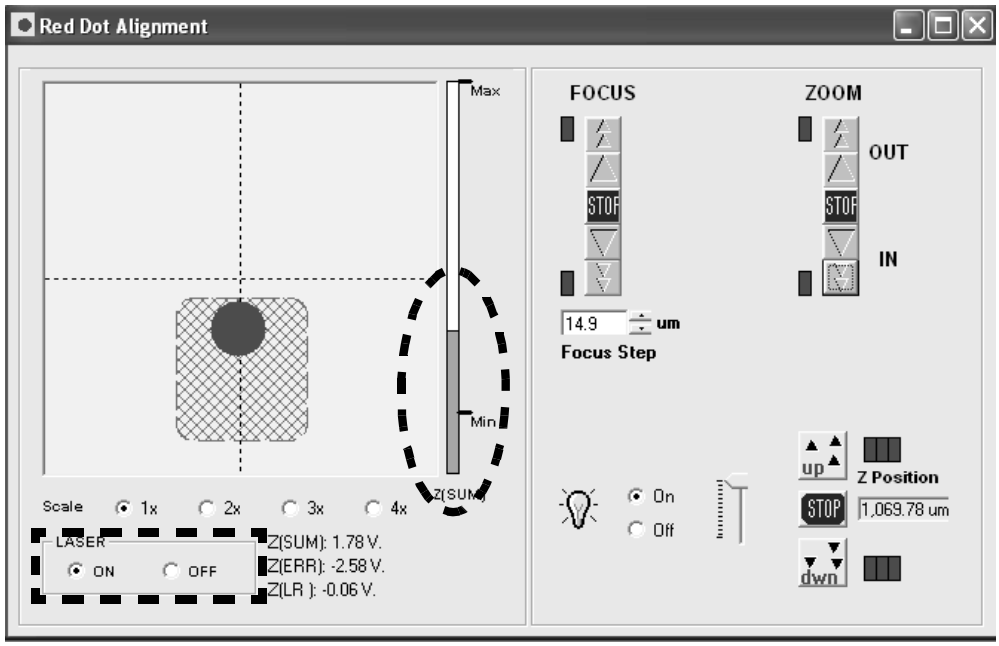


Figure 2.27. Aligning the detector.

7 Make sure the Z(SUM) value (signal intensity) is above the minimum.

If it is not, you need to re-seat or replace the probe.

APPROACHING THE SAMPLE

1 Click **Stage** from the EZMode toolbar.

2 Use the focus controls to bring the sample surface into focus on the video monitor.

CAUTION: Whenever you engage the motorized X-Y stage, be sure the probe is a safe distance above the sample/puck.

3 Use the X-Y stage controls to navigate to the largest of the four patterns on the PNI AFM reference (10 µm squares / 20 µm pitch).

Increase or decrease the X-Y step size, as desired, to facilitate both coarse and fine movements.

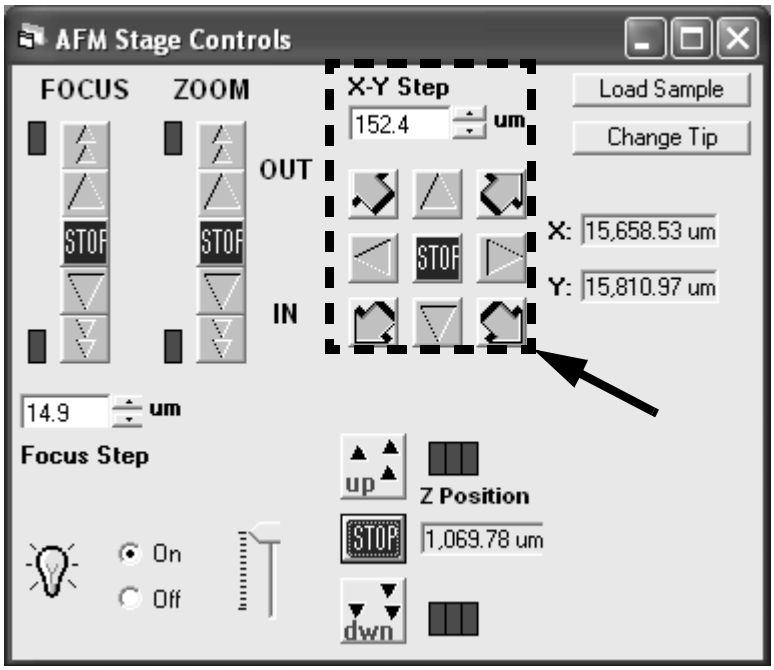


Figure 2.28. X-Y stage controls.

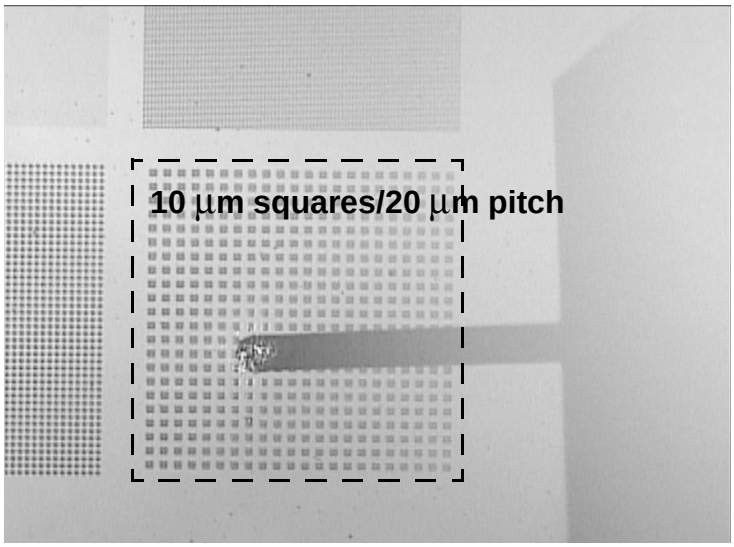


Figure 2.29. Positioning the probe over the scan area.

If necessary, you can orient the sample by simply rotating the puck by hand.

4 Focus on the cantilever.

CAUTION: Be careful not to drive the probe all the way into the sample surface.

5 While carefully monitoring the probe-sample distance by eye, use the 

button to lower the Z scanner until the probe is about 1-2 mm above the sample surface.

6 Focus on the sample surface, and make sure the probe is positioned somewhere near the center of the pattern.

7 Click the Tip Approach button on the toolbar.

8 Click OK when the tip approach is complete.

CAUTION: Once the tip approach is complete, and the tip is in contact with the sample surface, do not exit the SPM Cockpit software or turn off the Controller without first retracting the tip. Doing so may cause damage to the tip, scanner, and sample.

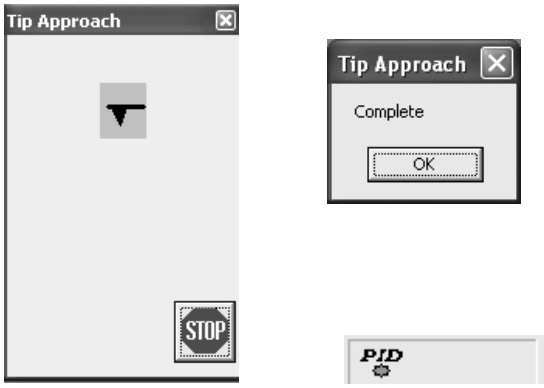


Figure 2.30. Tip approach confirmation.

The PID indicator at the bottom of the window will turn green to indicate that the probe tip is in contact with the sample surface, and the instrument is now ready to perform a scan.

SCAN THE SAMPLE

1 Click the Scan Sample button on the toolbar.

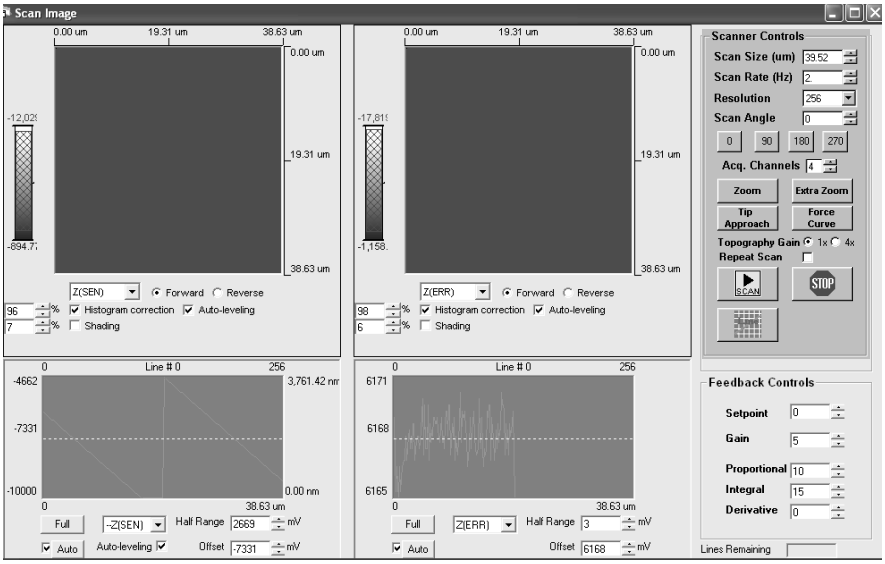
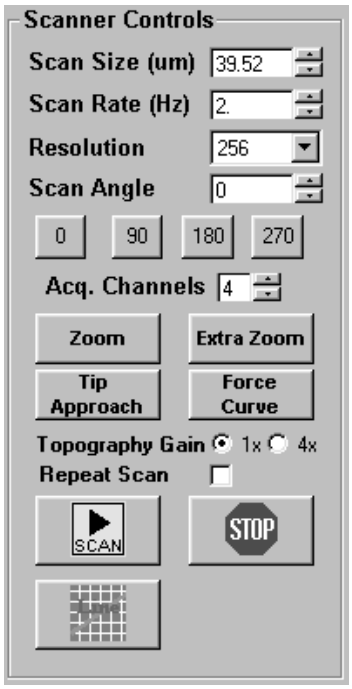


Figure 2.31. Scan image window.

2 Set the scanner controls as follows:

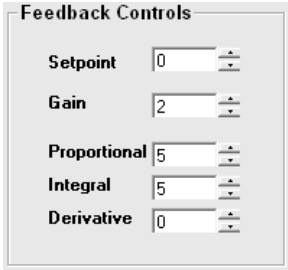
- Scan Size: leave as is*
- Scan Rage: 2 Hz
- Resolution: 256
- Scan Angle: 0
- Acq. Channels: 4
- Topography Gain: 1x

* The default scan size, which is entered by the system when the calibration routine is performed, is the maximum scan area for your scanner.



3 Set the feedback controls as follows:

- Setpoint: 0
- Gain: 2
- Proportional: 5
- Integral: 5
- Derivative: 0



4 Select the Z(SEN) and Z(ERR) channels from the drop-down menus beneath the two image displays, and for each display, select Forward, Histogram-correction, and Auto-leveling.

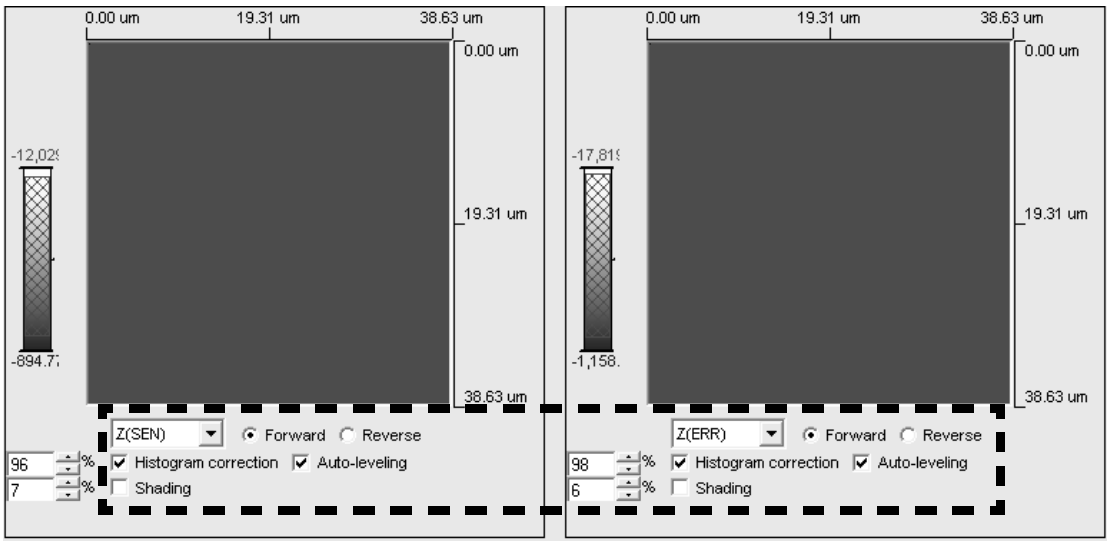


Figure 2.32. Image display settings.

5 Select the Z(SEN) and Z(ERR) channels from the drop-down menus of the two corresponding line scan displays.

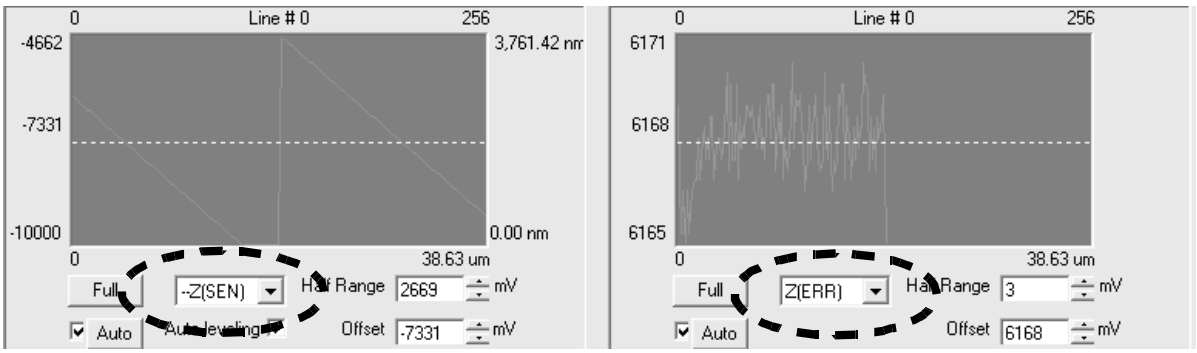


Figure 2.33. Line scan settings.

6 Click  to take a scan.

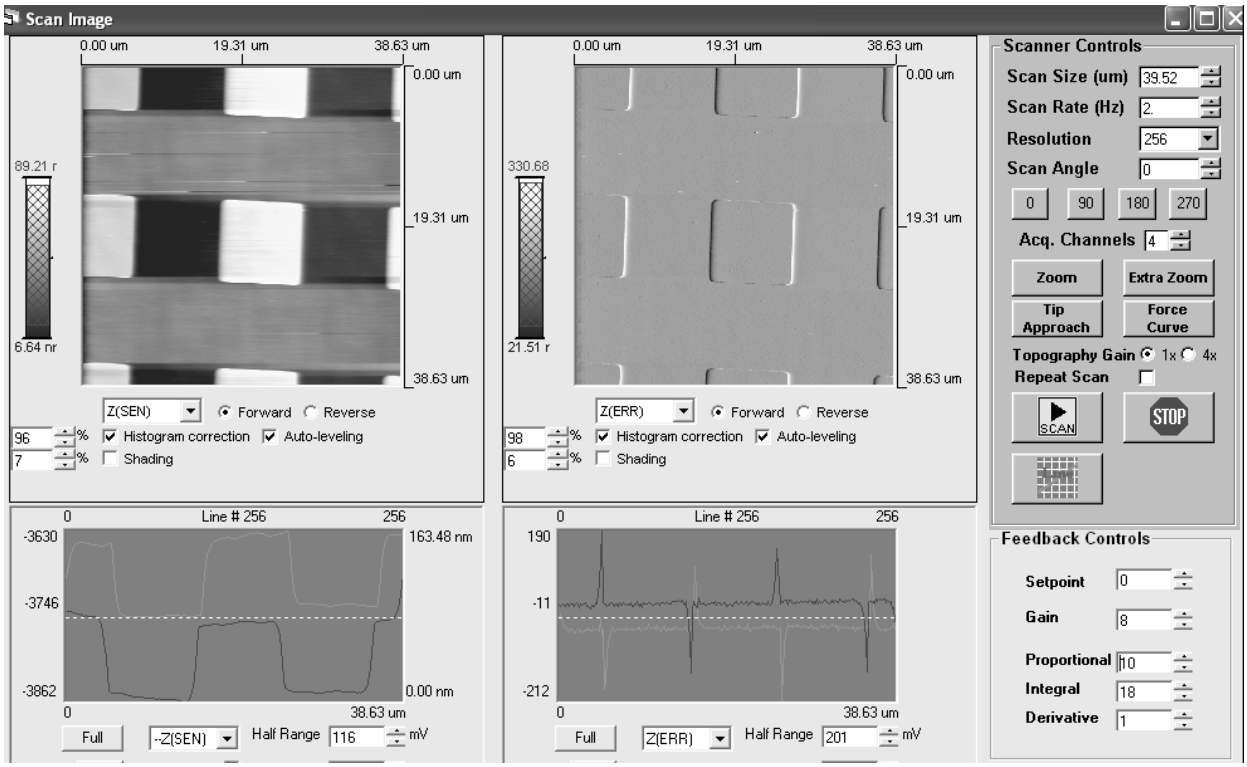
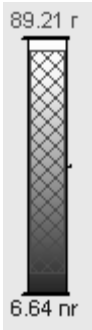


Figure 2.34. Taking a scan.

- The images of the selected channels will build up line-by-line in the displays. If no data is generated, the detector may be out of alignment. In this case, click the STOP button, click Tip Retract from the toolbar, re-align the red dot (page 23), and try another scan.
- To adjust the Z scale of the images, left-click and drag in the bar to the left of each display to select a Z height range.
- To view a single line scan, hold down the SHIFT key and left-click in either image display to define a horizontal line across the image; make sure the line includes the square features. The line scan profile for the Z(SEN) channel should resemble the shape and size of the 10 µm features.



Z scale adjust

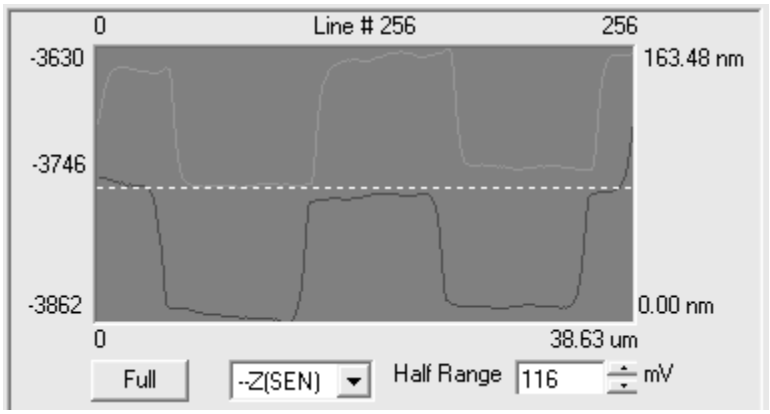


Figure 2.35. Viewing a single line scan.

When the feedback controls are properly set, the forward and reverse line scan profiles for the Z(ERR) channel will roughly mirror each other.

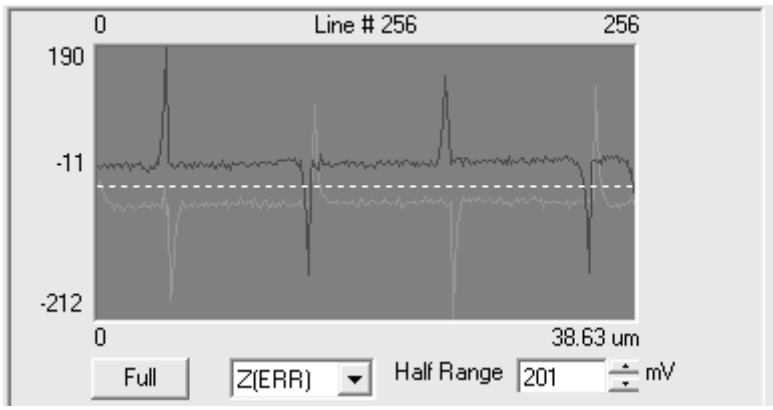


Figure 2.36. Z(ERR) channel line profile.

7 To take additional scans, click SCAN again, or check Repeat Scan to take continuous scans of the same region.

8 To zoom to a new scan region:

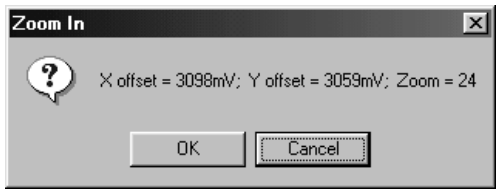
a) Left-click and drag in the image display to define a scan area.

b) Left-click again to position the box within the scan region.

c) Right-click to confirm the new scan region.

d) Click OK to zoom to the new scan area.

The probe will move to the new scan region, where you can start a new scan. Additional zoom features are accessible via the Zoom and Extra Zoom buttons, or by simply double-clicking in the display.



Zoom confirm

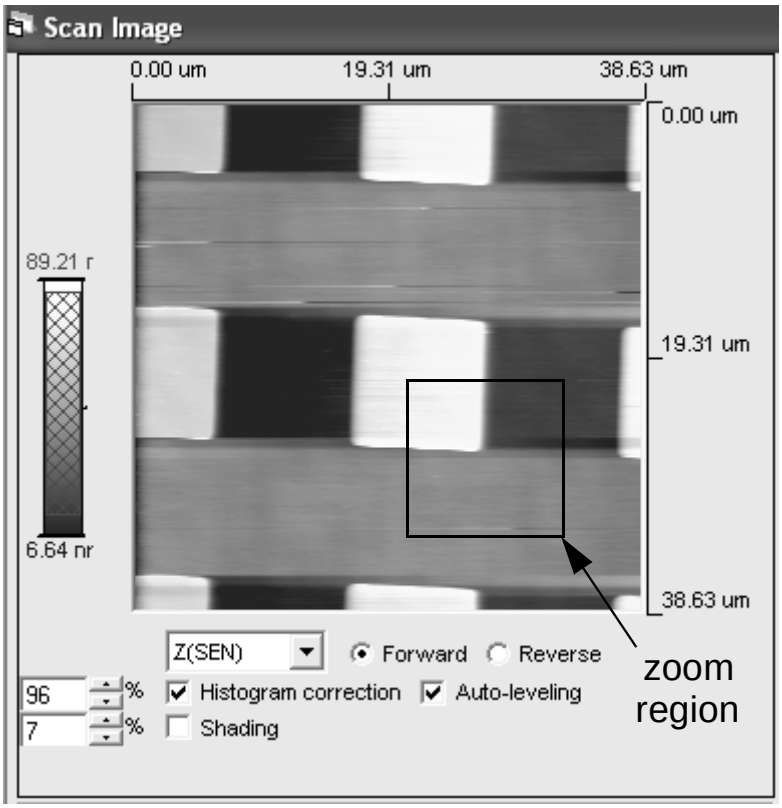


Figure 2.37. Selecting a zoomed-in scan region.

9 To end your session now, click Tip Retract on the EZMode toolbar.

Once the tip is retracted, it is safe to turn off the Master Computer and the Controller.

IMAGE PROCESSING

1 Click Image Processing on the EZMode tool bar.

2 Click  to open an image for processing.

Figure 2.38. Image processing module.

3 Select the desired acquisition channel and direction for the image to be processed.

The raw image data will not resemble the image in the scan image window, as some basic real-time image processing was applied as it was being acquired.

4 Click  to apply a plane correction.

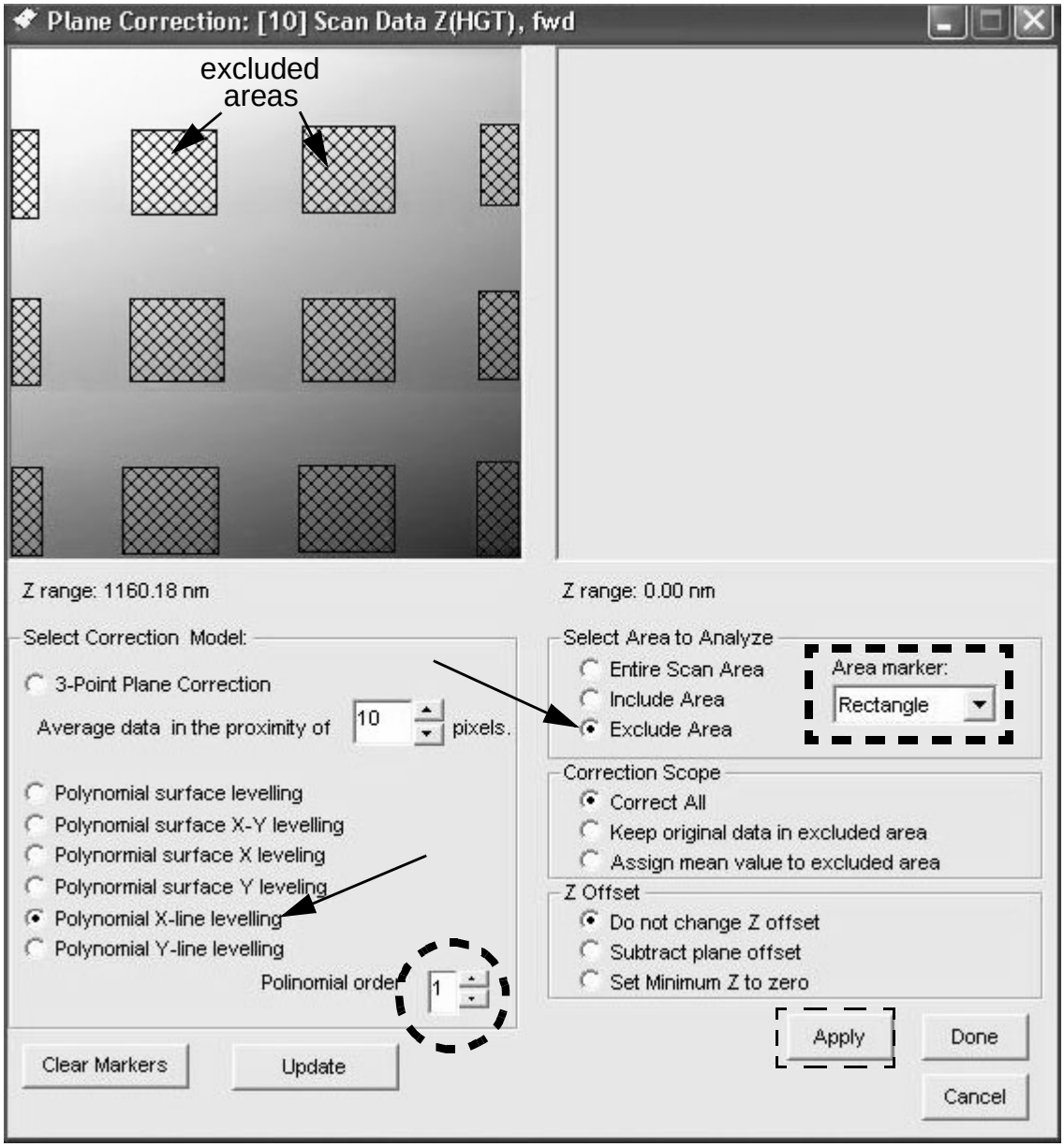


Figure 2.39. Plane leveling tool.

a) Under Select Correction Model, select:
- Polynomial X-line leveling
- Polynomial order: 1

b) Under Select Area to Analyze, select:
- Exclude Area
- Area marker: Rectangle.

c) To exclude the features on the PNI AFM reference, use the mouse to left-click and drag in the image display so that every feature (both whole and partial) is completely covered.

d) Click Apply, and the leveled image appears in the right-hand display.

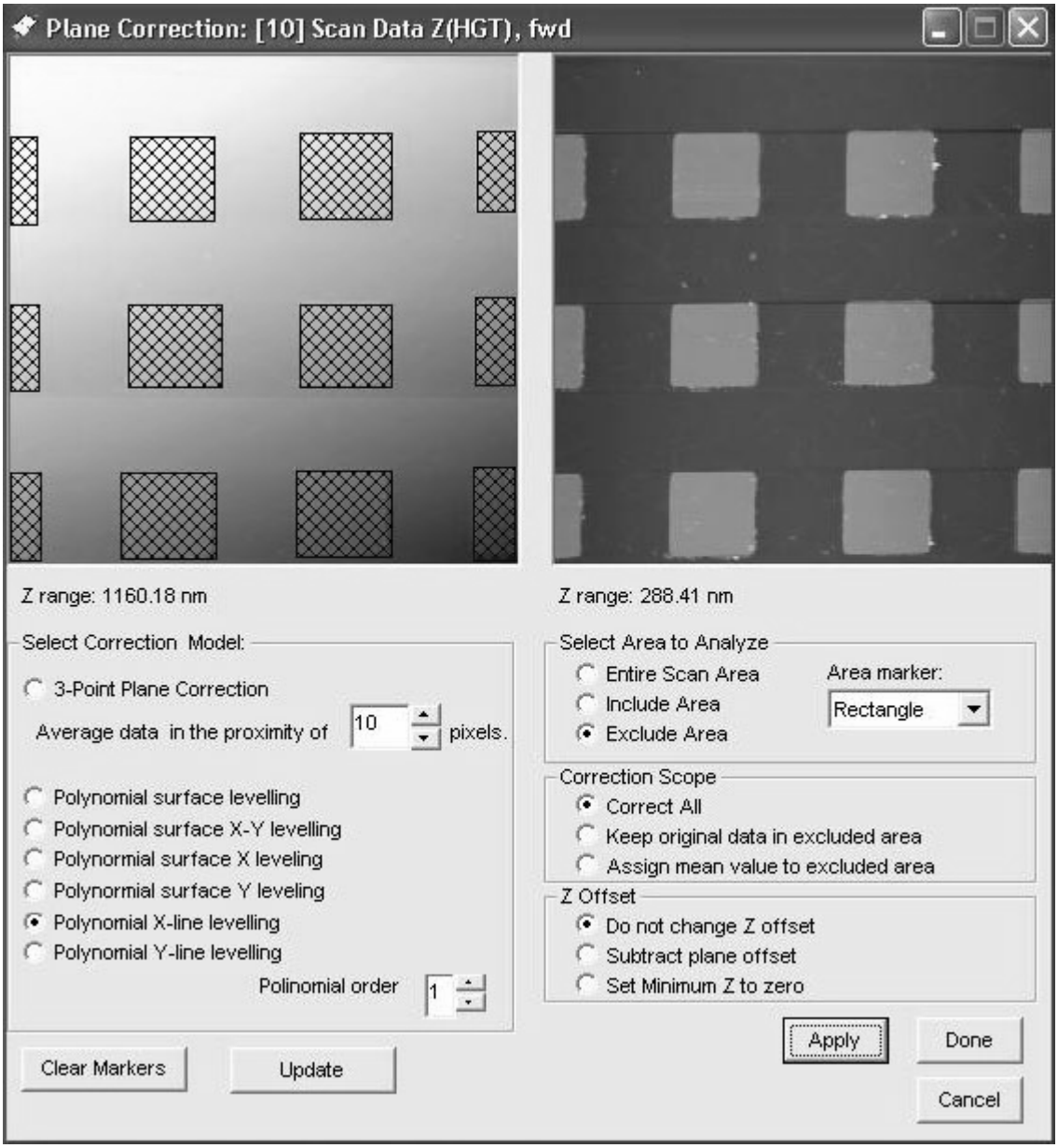


Figure 2.40. Leveled image (right).

5 Click  to open the line profile tool.

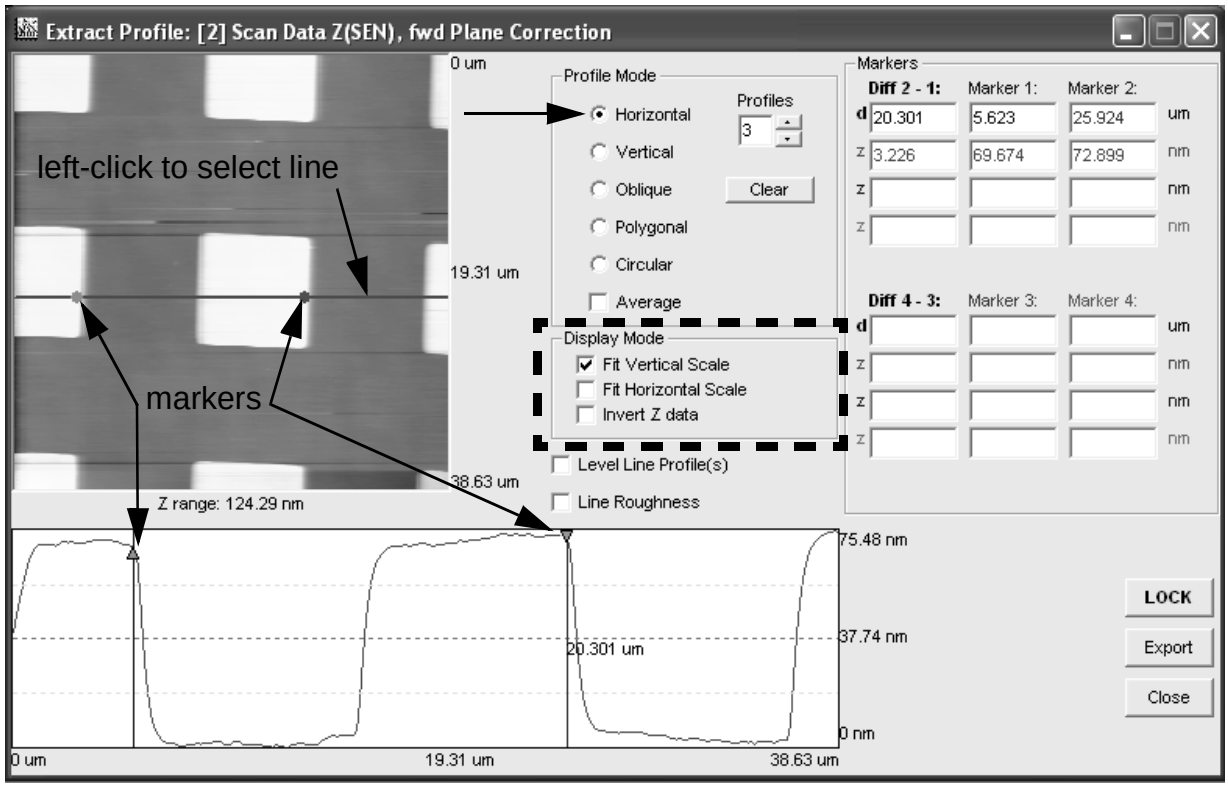


Figure 2.41. Line profile tool.

a) Under Profile Mode, select Horizontal.

b) Under Display Mode:

- Check Fit Vertical Scale
- Uncheck Invert Data

c) Left-click in the image display to select a line.

d) Left-click in the line display to make measurement markers.

In the example above, measurements are made between the edges of two consecutive features on the PNI AFM reference. The measurements displayed to the right confirm a pitch of 20 µm and a Z-height of approximately 70 nm.

NOTE: These measurements should not be used to calibrate your instrument!

6 Click  to open the histogram tool, and use the slider bars to mark the middle of the two ranges where the Z data points are clustered.

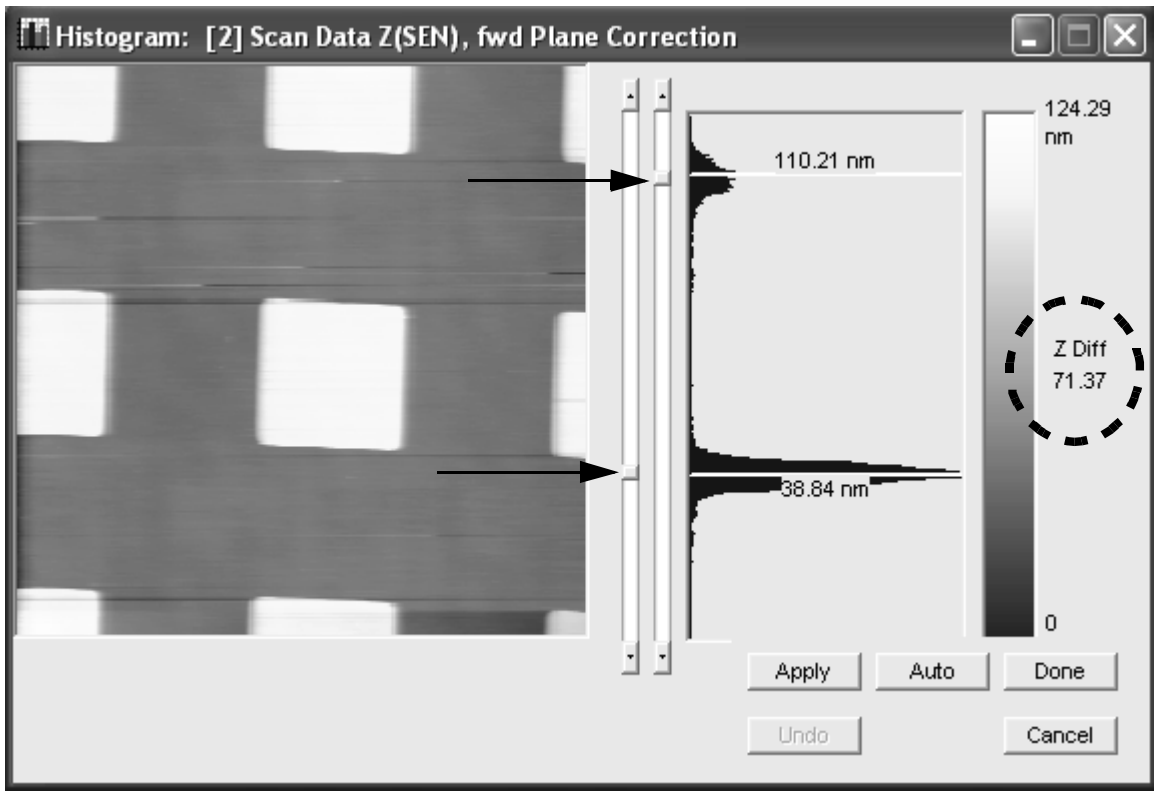


Figure 2.42. Histogram tool.

The Z Diff measurement on the vertical bar confirms the 70 nm height of the PNI AFM reference features.

7 To save any of your processed images, select File→Save Image(s).

8 Click  to return to the acquisition module.

CAUTION: To prevent damage to your scanner, probe, and sample, be sure to retract the tip before exiting the SPM Cockpit software or turning off the Controller.

9 Click Tip Retract.

It is now safe to exit the SPM Cockpit software.

Chapter 3

Tutorial: Close Contact EZMode

BEFORE YOU BEGIN

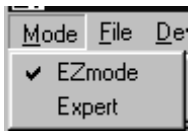
This tutorial follows the steps for an taking a contact AFM image of the PNI AFM reference in EZMode.

WARNING: *Before operating the Nano-R AFM, make sure you are familiar with the safety information on page vi.*

POWERING UP THE SYSTEM

1. Turn on the Master Computer.
2. Launch the SPM Cockpit software.
3. Turn on the Controller.
4. Turn on the video monitor.

SOFTWARE SETUP



1. Select Mode→EZMode.
2. Click the Start button from the EZMode toolbar.

3 Click Retract Tip, and click OK when complete.

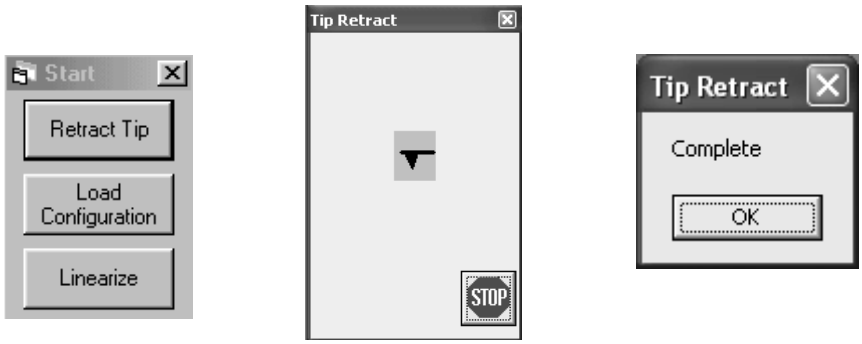


Figure 3.1. Retracting the tip.

4 Click Load Configuration, select the PNI-supplied close-contact mode configuration file, and click Open.

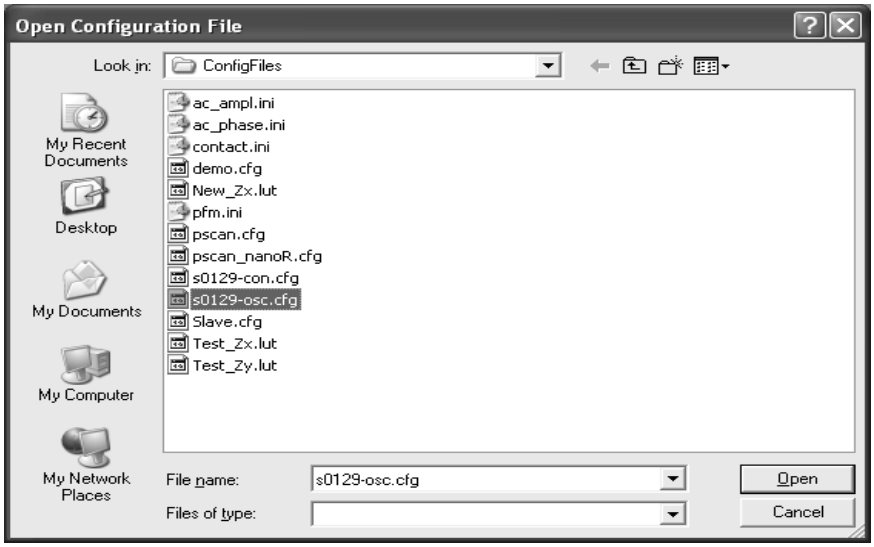


Figure 3.2. Loading a configuration file.

This file should be located in the ConfigFiles folder in the SPM Cockpit directory. It has the format sxxxx-osc.cfg, where xxxx is the serial number of your Nano-R system.

5 Click Linearize, check both boxes, and click OK.

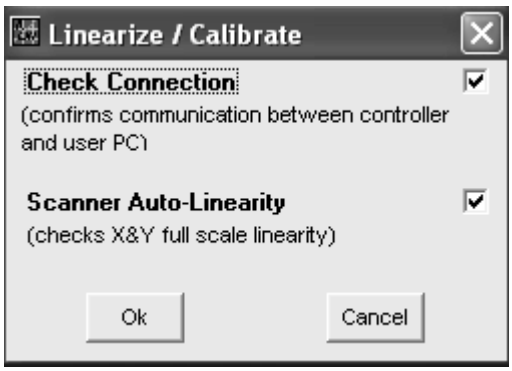


Figure 3.3. Initiate connection confirm and calibration routines.

6 Click OK when the communication between the Master Computer and the Controller is confirmed.

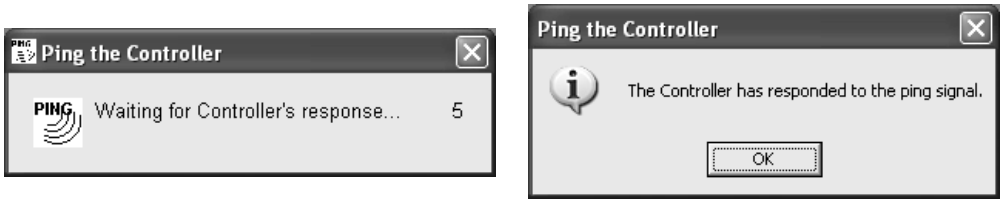


Figure 3.4. Connection confirmed.

If there is no connection, you need to exit the SPM Cockpit software and restart both the Master Computer and the Controller.

7 Click Yes to proceed with the calibration procedure.

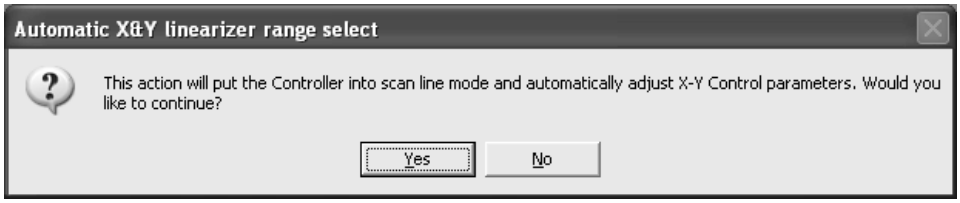


Figure 3.5. Proceed with calibration routine.

8 Click OK when the last step of the calibration process is complete, and then click OK to proceed.

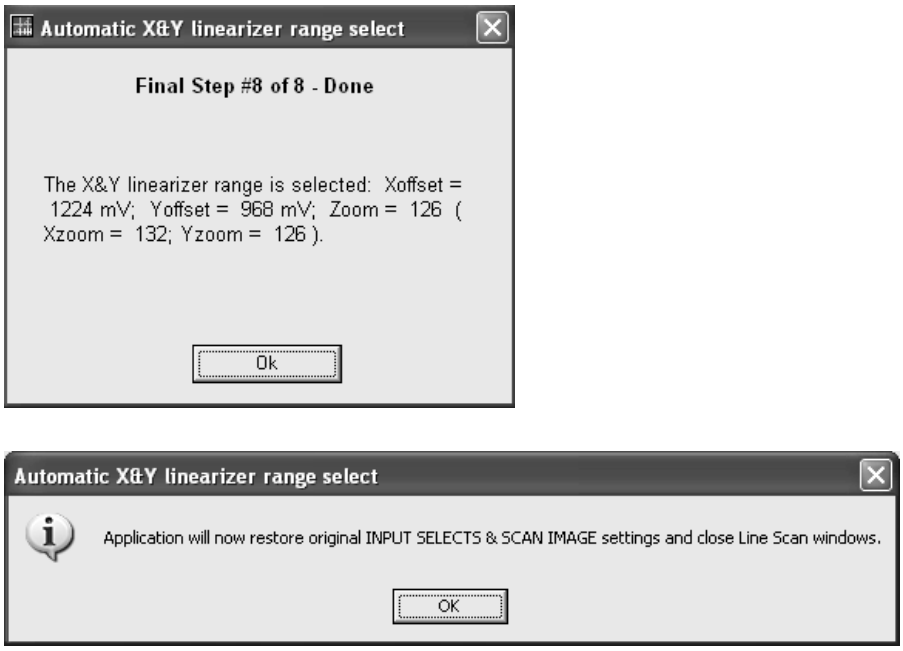


Figure 3.6. Calibration routine complete.

9 Click Select Mode on the toolbar, select Close Contact, and click OK.

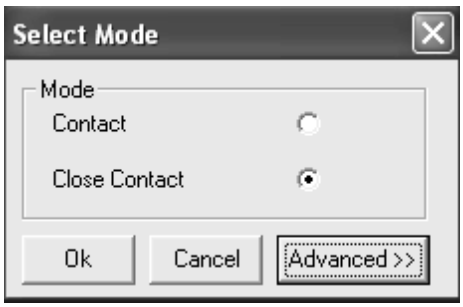


Figure 3.7. Select mode.

10 If the PNI AFM reference is already loaded on the sample puck, skip ahead to page 45 to load a probe, or to page 52 if a contact probe is already loaded.

LOAD A SAMPLE

1 Click Tip Retract from the EZMode toolbar.

2 Click Stage from the EZMode toolbar.

3 Click 

to raise the Z motor until there is at least a few millimeters of clearance between the probe and the sample surface (monitor by eye).

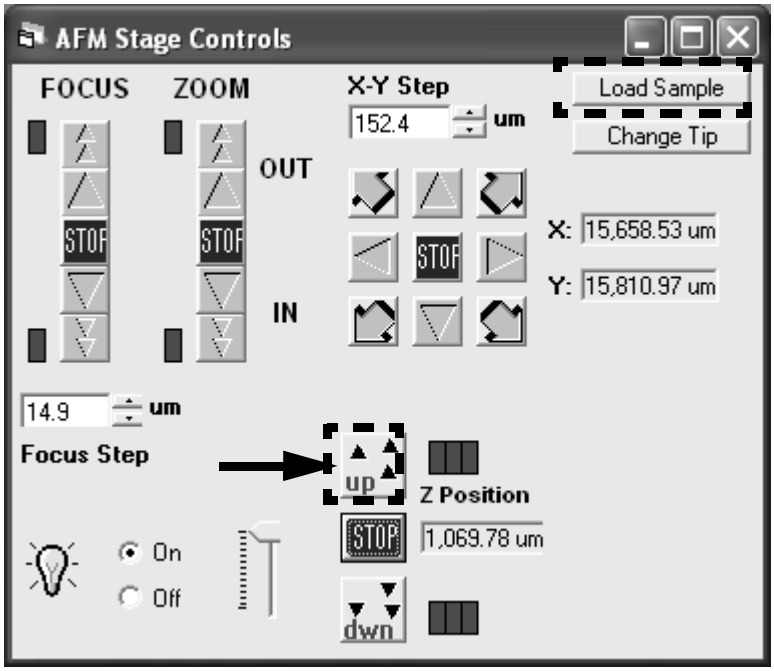


Figure 3.8. Raise the probe tip away from the sample.

CAUTION: To prevent damage to your scanner, probe, and sample, be sure you have retracted the tip and raised the Z scanner (as described in the preceding steps) before moving the puck.

4 Click 

.

The motorized X-Y stage will move the puck towards you, to the limit of its range.

5 Being careful not to touch the probe, slide the sample puck towards you, and lift it up out of the groove.

6 Use tweezers to mount the PNI AFM reference on the center of the puck.

The sample disk is held in place magnetically.

7 Replace the puck on the stage by setting it down so the protruding piece on the bottom fits into the wide part of the groove, and then slide it into position.

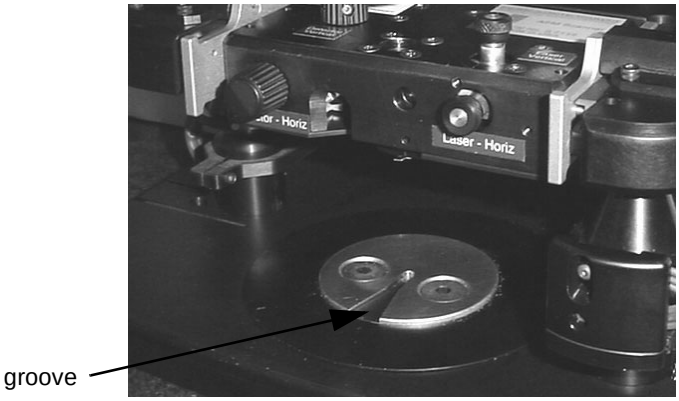


Figure 3.9. Fit the sample puck into the groove on the X-Y stage.

8 Rotate the puck so that the PNI reference sample is square with the scanner head.

9 Select Tools→NanoR Stage to open the AFM Stage Controls window.

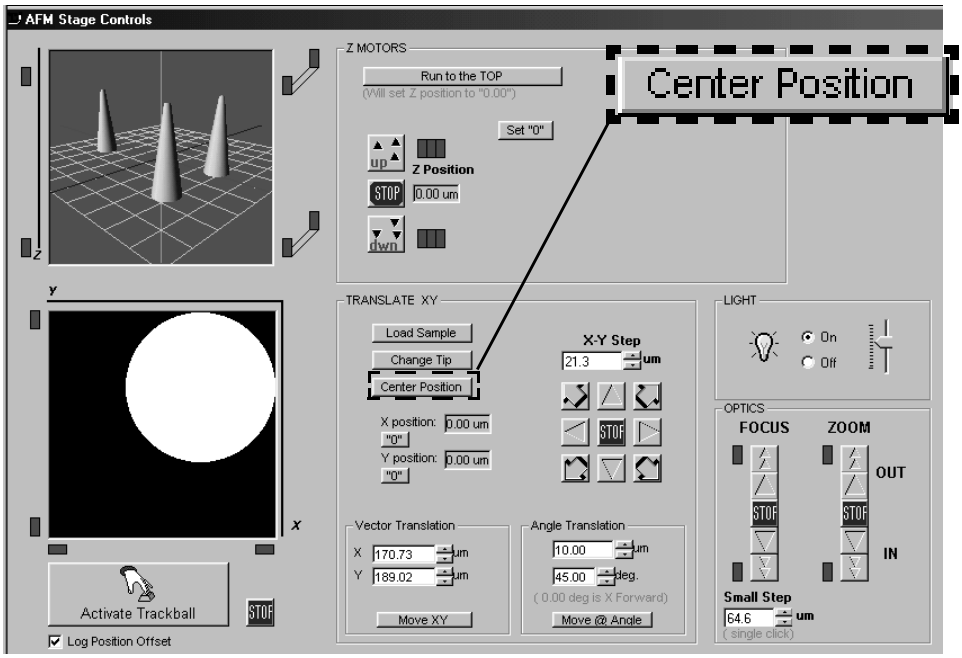


Figure 3.10. Return sample puck to initial X-Y position.

10 Click Center Position.

The motorized X-Y stage will return the puck to its original position

INSTALL A PROBE

To operate in close-contact mode, you need to use a close-contact probe. Probes should be stored in the supplied boxes, which are marked "Contact" and "Close-contact," as the differences between the two types of probes is not visible to the naked eye.

1 First, remove the sample puck as described in the section above.

2 Click Stage from the EZMode toolbar, and click Change Tip in the AFM Stage Controls dialog.

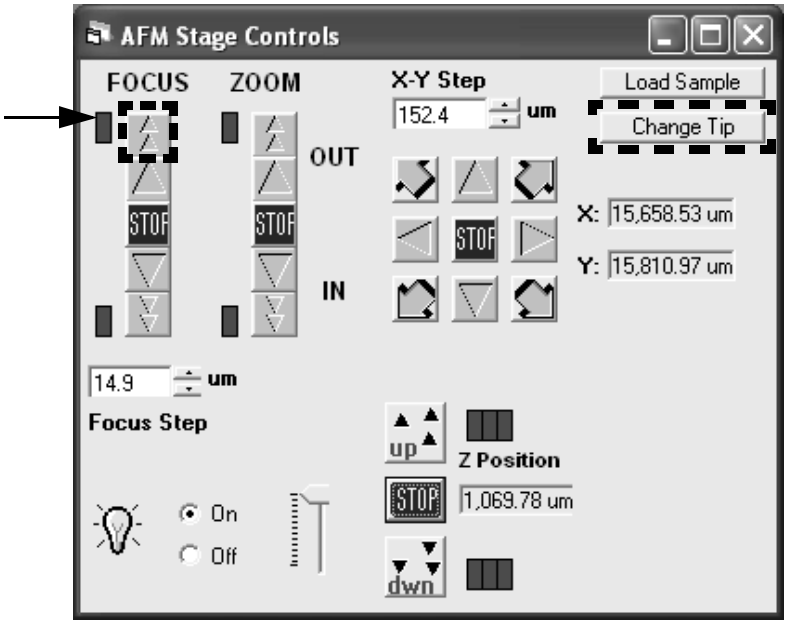


Figure 3.11. Raise the probe tip away from the sample.

The Z motors will raise the scanner to the top of its range.

3 Click the focus button to raise the video objective to the top of its range. The upper indicator will turn from green to red when the objective reaches the top of its range.

4 Click Align Laser from the tool bar.

5 Turn off the laser.

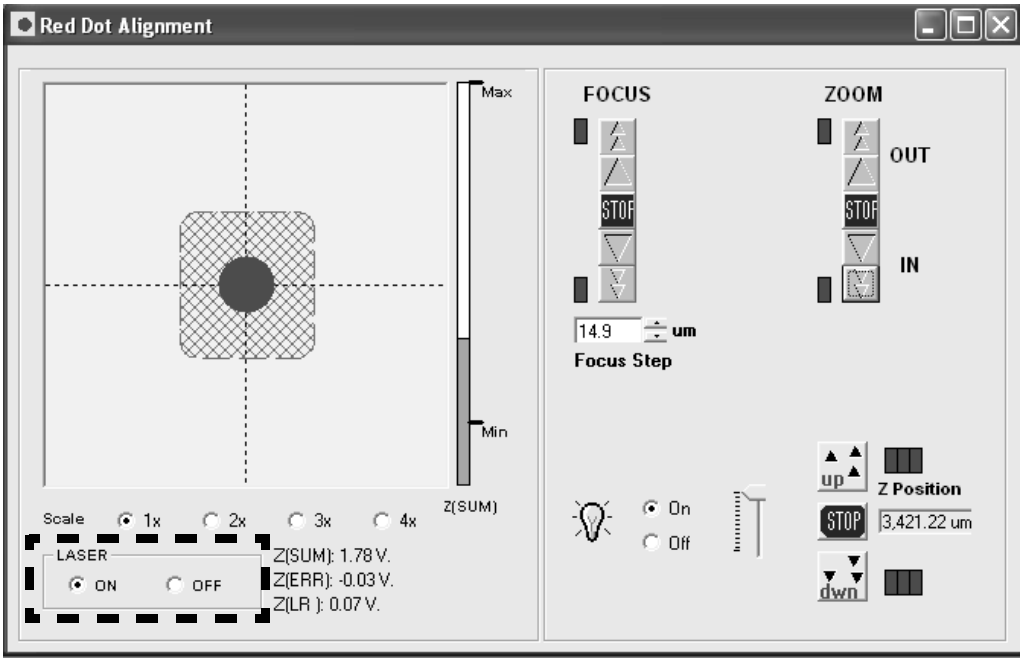


Figure 3.12. Turning off the laser.

WARNING: *To avoid potentially hazardous laser exposure, be sure to turn off the laser before rotating the scanner into the probe exchange position.*

6 Turn the probe exchange knobs on the side of the scanner head down (away from you) 1/4 turn (Figure 3.13).

The scanner head will slide out about an inch.

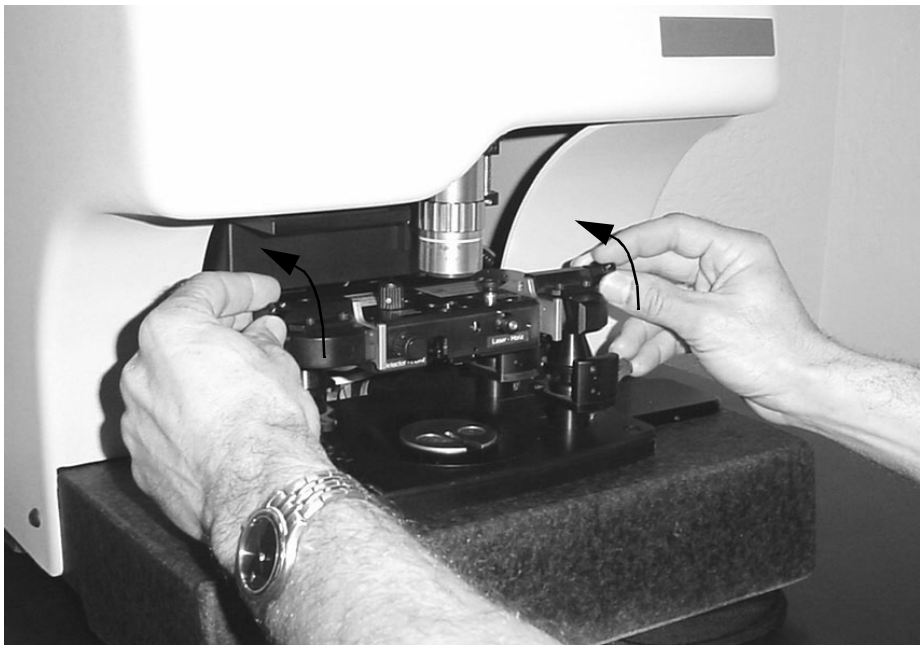


Figure 3.13. Turn the knobs to disengage the scanner head.

7 Grasp the handles on the front of the scanner head (Figure 3.14), and gently slide the scanner head all the way towards you.

Figure 3.14. Slide the scanner head toward you.

8 Carefully rotate the scanner head up about 90 degrees, as shown in Figure 3.15.

Figure 3.15. Rotate the scanner head.

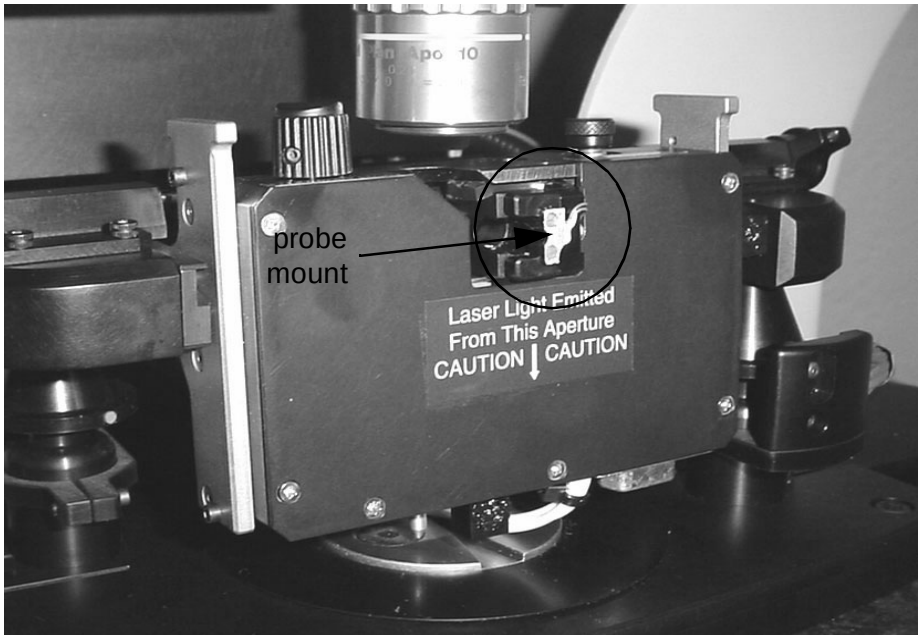


Figure 3.16. Probe exchange position.

CAUTION: Handle AFM probes with care. The cantilever can break off easily if it touches anything or snaps down too forcefully on the magnetic mounting surface on either the scanner or in the probe box.

Probe handling: When loading or removing a probe, pivot the substrate on the edge opposite the cantilever, as shown in Figure 3.17. This will protect the cantilever from striking the magnetic mounting surface, and it will prevent the substrate from snapping down too forcefully, which may damage the probe.

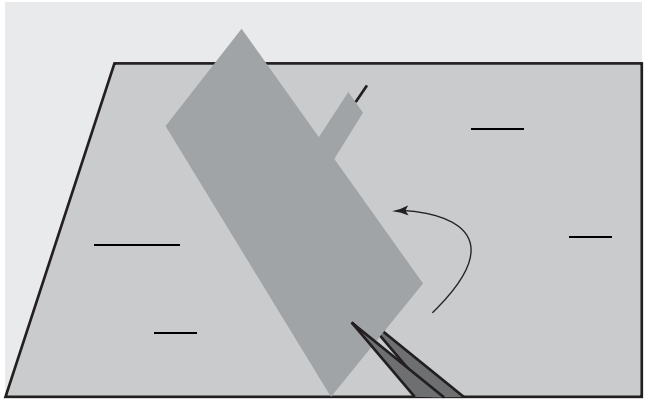


Figure 3.17. Probe handling.

9 To remove a probe:

- **a)** Use tweezers to grasp the metal substrate as indicated in Figure 3.20.
- **b)** Carefully rotate the tweezers so the cantilever side of the substrate lifts up off the magnetic mount first.
- **c)** Set the probe down onto the magnetic strip in the probe box so that the side of the substrate opposite the cantilever makes contact first.
- **d)** Carefully rotate the tweezers so the cantilever side of the substrate comes down onto the magnetic surface as gently as possible.

10 Apply a small amount of glycerol to the probe mount.

This will ensure proper mechanical coupling between the probe and the mount, which is essential for close-contact (vibrating cantilever) operation.

11 To install a new probe:

- **a)** Use tweezers to nudge a probe so that the substrate extends over the edge of the magnetic strip in the probe box (Figure 3.18).
- **b)** Grasp the metal substrate, and carefully rotate the tweezers so the cantilever side of the substrate lifts up off the magnetic strip first, as shown in Figure 3.19.

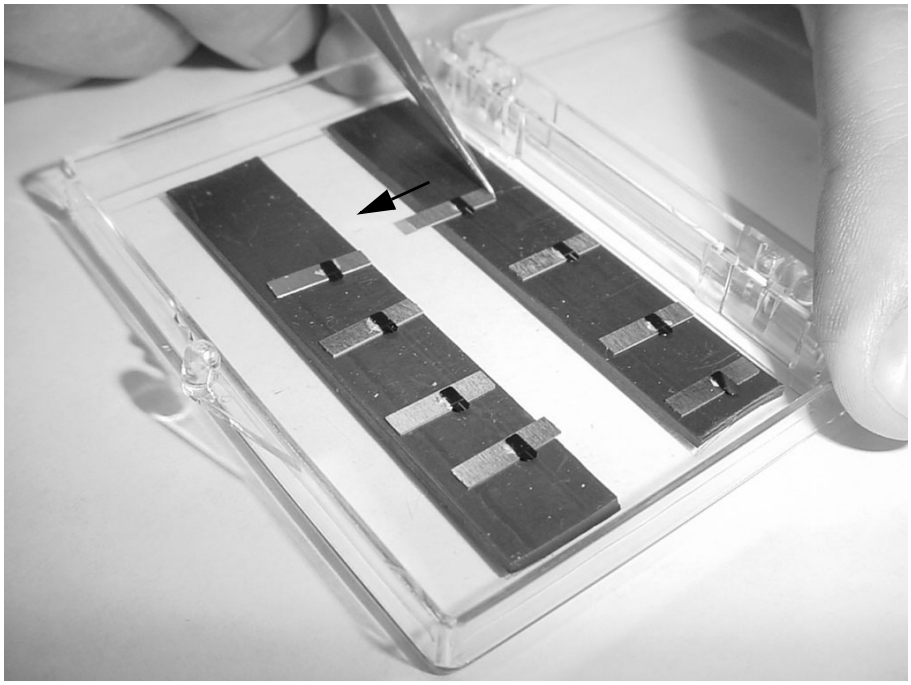


Figure 3.18. Nudge the probe into position.

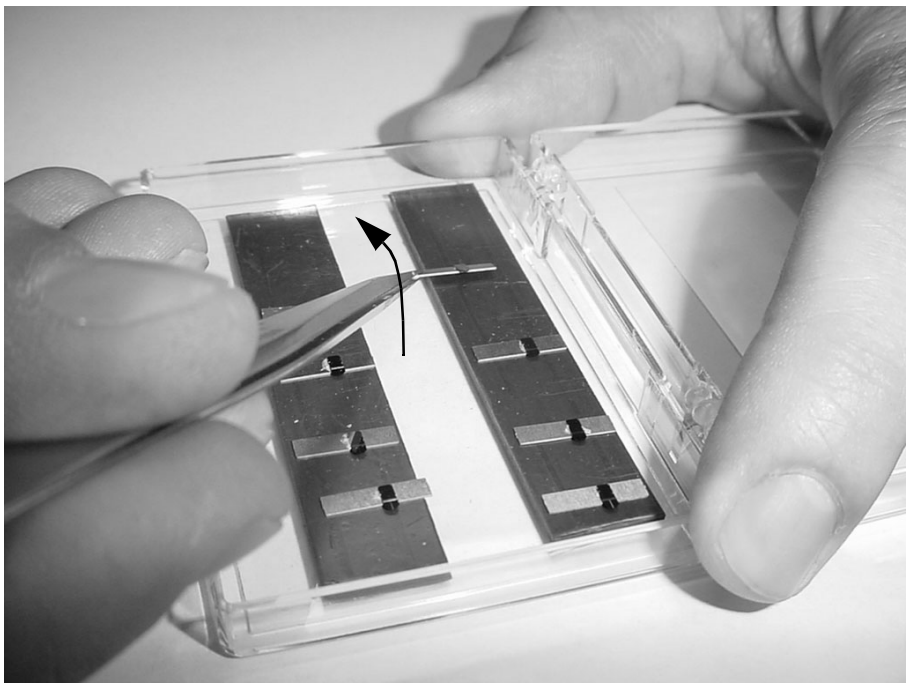


Figure 3.19. Lift the probe, cantilever side first.

c) Place the probe onto the magnetic mount so the side of the substrate opposite the cantilever fits into the "L."

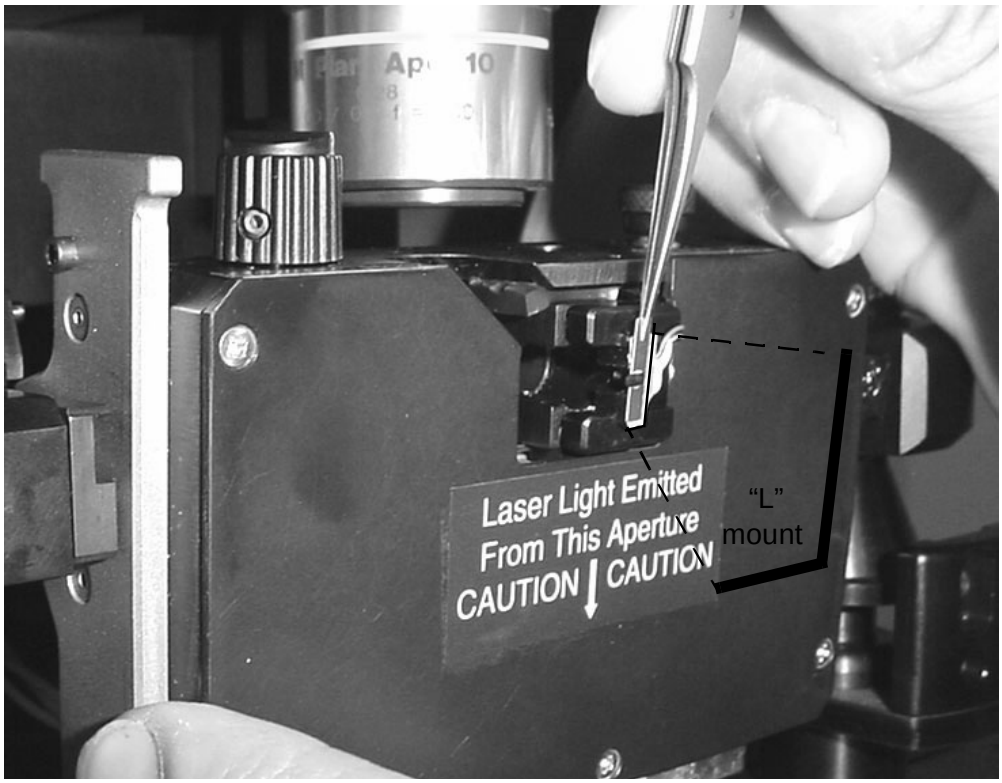


Figure 3.20. Mounting the probe.

d) Use the tweezers to push the substrate flush against the "L," as shown in Figure 3.21.

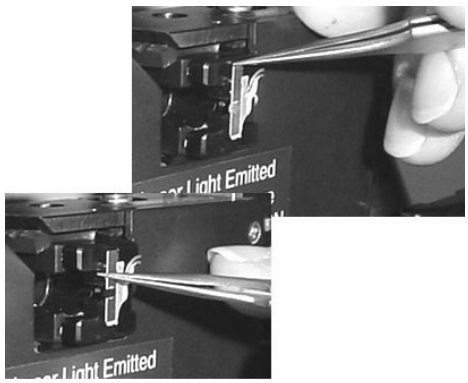


Figure 3.21. Push the probe substrate flush against the "L" mount.

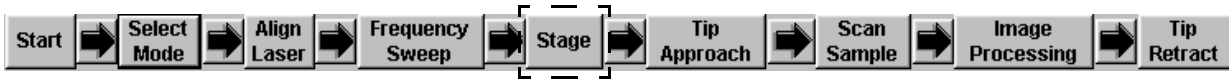
12 Hold the scanner head by the handles, and rotate it back to the level position.

13 Gently slide the scanner back towards the stage until you feel some resistance.

14 Turn the probe exchange knobs up (1/4 turn) to lock the scanner head into place.

Now you can replace the sample puck, as described above (page 44).

ALIGN THE DETECTOR



1 Click Stage from the EZMode toolbar.

2 Use the focus controls to bring the probe tip into focus on the video monitor.

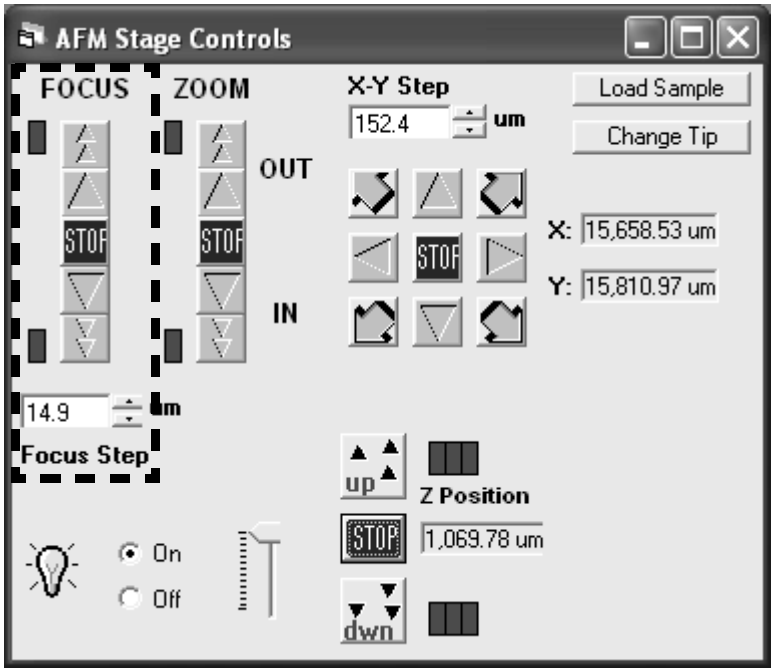


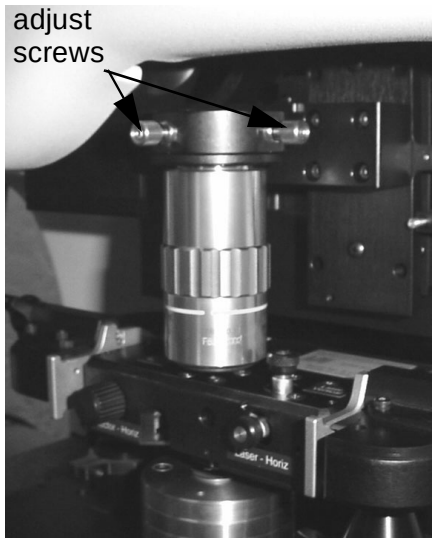
Figure 3.22. Focus controls.

Focus controls: Click  to adjust the focus a single step, or hold it down for continuous motion. Click  to initiate a large, continuous movement of pre-set duration.

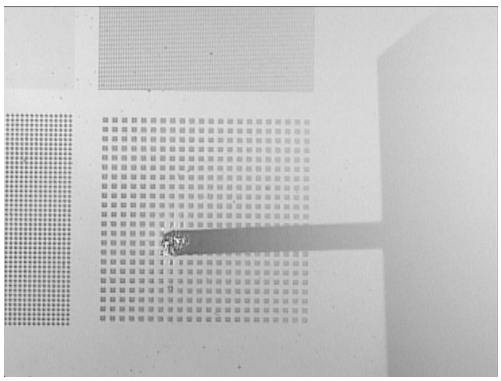
If you cannot find the probe on the monitor:

- The probe may not have been installed properly. Repeat the probe installation procedure to make sure the probe is seated squarely in the "L" mount (page 51).
- The objective's field of view may need to be adjusted in X-Y, using the adjust screws. This is usually necessary when switching between a contact and close-contact probe, due to the difference in size.

You can confirm that you have installed a contact cantilever by noting the difference in length between contact and close-contact cantilevers, as shown in Figure 3.25.



close-contact cantilever



contact cantilever

Figure 3.23. Contact vs. close-contact probes.

3 Click Scan Sample from the toolbar, and make sure the Setpoint value in the Feedback Controls box is 0.

4 Click Align Laser on the EZMode toolbar.

5 Turn on the laser.

The red dot alignment procedure has 3 goals:

- position the laser spot on the back of the cantilever
- position the photodetector in the center of the reflected laser beam
- achieve a minimum overall measured signal strength

6 Watch the video monitor as you adjust the laser alignment knobs on the scanner head to bring the laser spot onto the back of the cantilever.

The laser spot should be centered on the cantilever, not too close to the end, as shown in Figure 3.24.

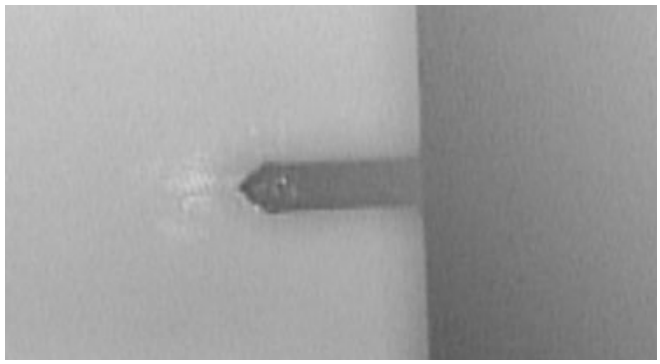


Figure 3.24. Centering the laser spot on the cantilever.

7 Watch the red dot (in the Red Dot Alignment window) as you turn the detector alignment knobs to bring the red dot into the center of the green box, as shown in Figure 3.25.

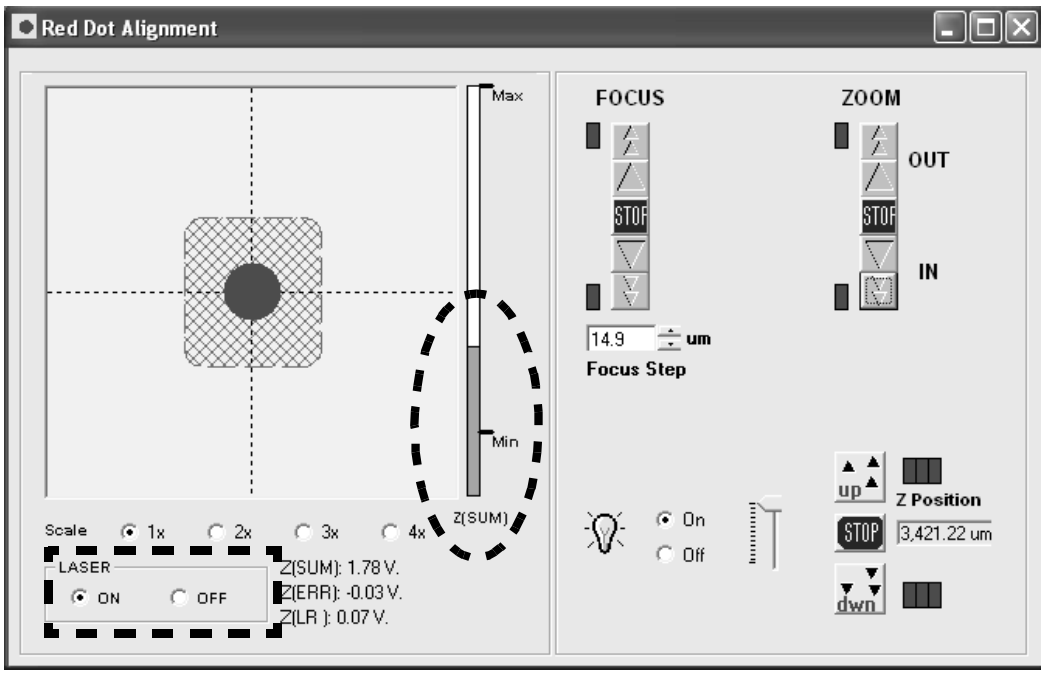


Figure 3.25. Aligning the detector.

8 Make sure the Z(SUM) value (signal intensity) is above the minimum.

If it is not, you need to re-seat or replace the probe.

FREQUENCY SWEEP

After aligning the detector, the resonant frequency for the installed cantilever must be set.

1 Click Frequency Sweep on the EZMode tool bar to open the Frequency sweep window.

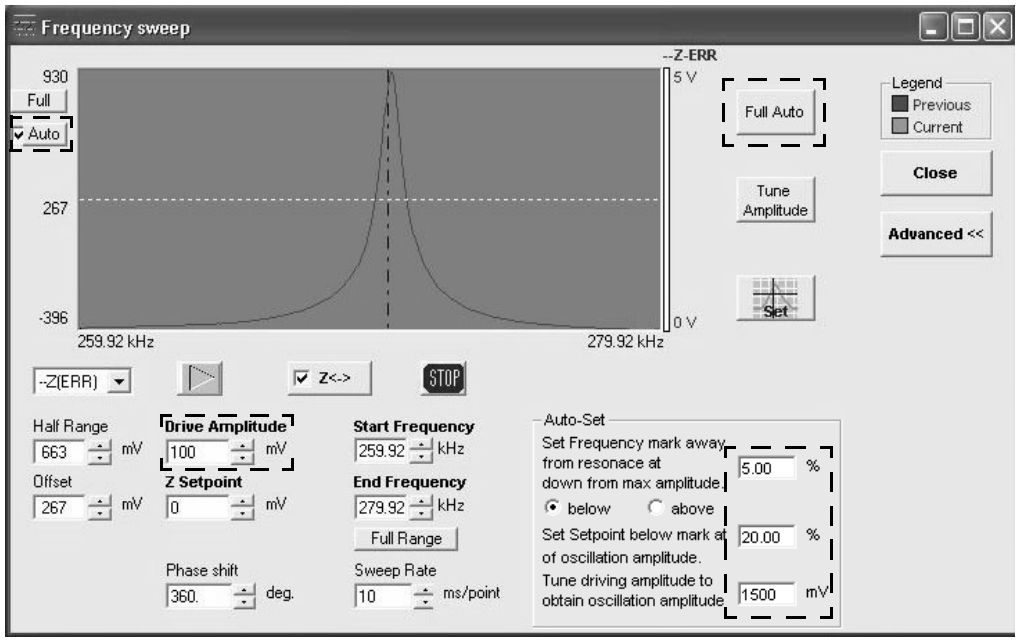


Figure 3.26. Frequency sweep window.

2 Set the Drive Amplitude to 100.

3 Make sure the Z Setpoint is 0.

4 Make sure the Auto Set values are set as follows:

- "Set Frequency mark...": 5.0%
- "Set Setpoint below mark...": 20.0%
- "Tune driving amplitude to...": 1500 mV

5 Check the Auto option.

6 Click Full Auto.

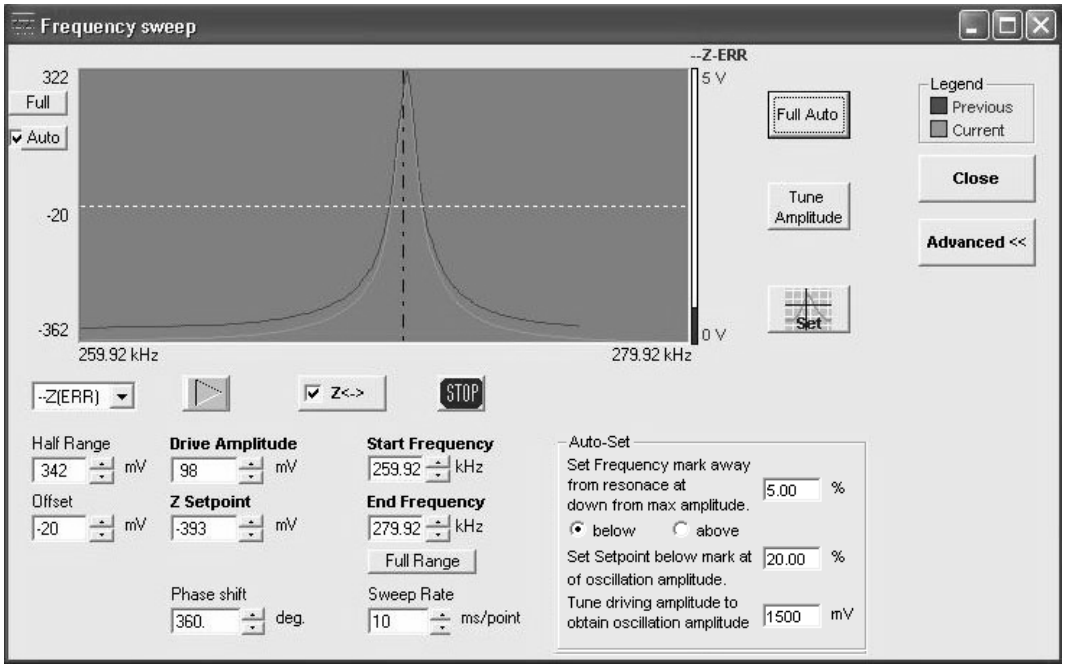


Figure 3.27. Initial frequency peak.

7 When the sweep is complete, click Tune Amplitude.

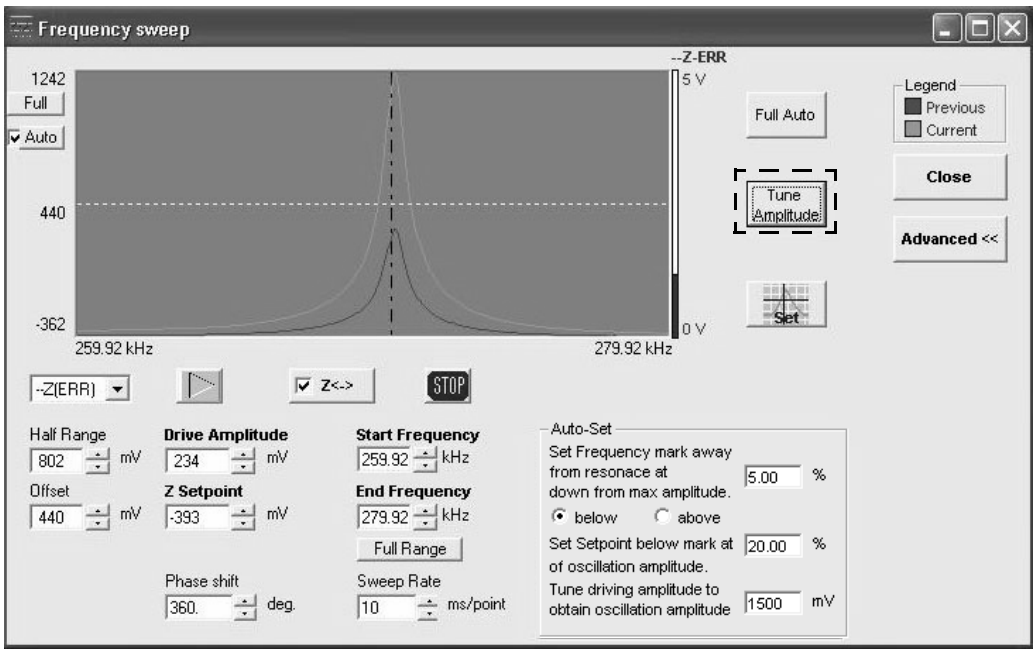


Figure 3.28. Tuned frequency peak.

8 Check the quality of the peak, and repeat the frequency sweep if necessary.

The resulting peak should be clean and sharp, as shown in Figure 3.28. If the line is noisy, or there are multiple peaks, it probably means the contact between the probe and the scanner is faulty. To remedy this, repeat the procedure for installing a probe, making sure that the probe substrate is flush with the "L" shaped mount. If you have not already done so, remove the probe, apply a small droplet of glycerol to the probe mount, and then install the probe.

9 Click 

to accept the selected peak.

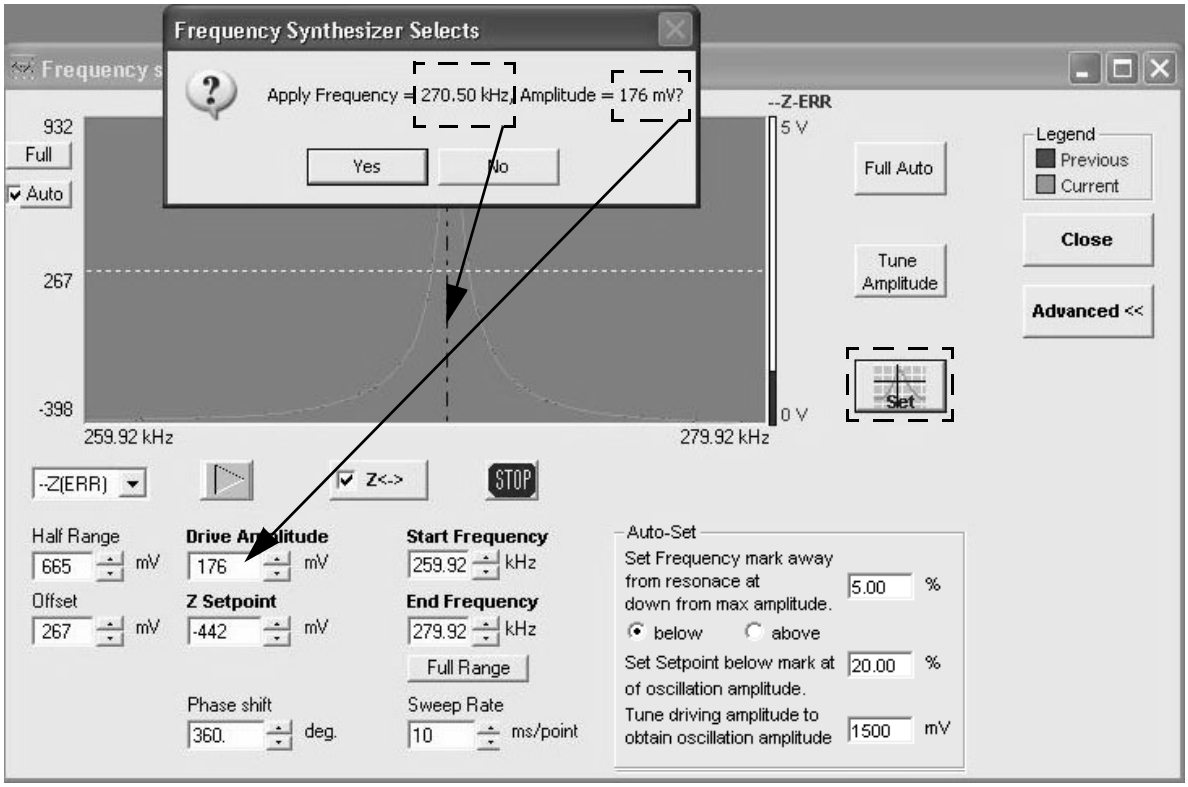


Figure 3.29. Confirm the frequency and drive amplitude.

10 Confirm that the frequency and amplitude values correspond to the values in the Frequency sweep window, and click Yes.

11 Click Yes in the Auto Setpoint Value confirmation box.

The system will enter the new setpoint value in the Z Setpoint field as well as in the Setpoint field in the Scan Image window.

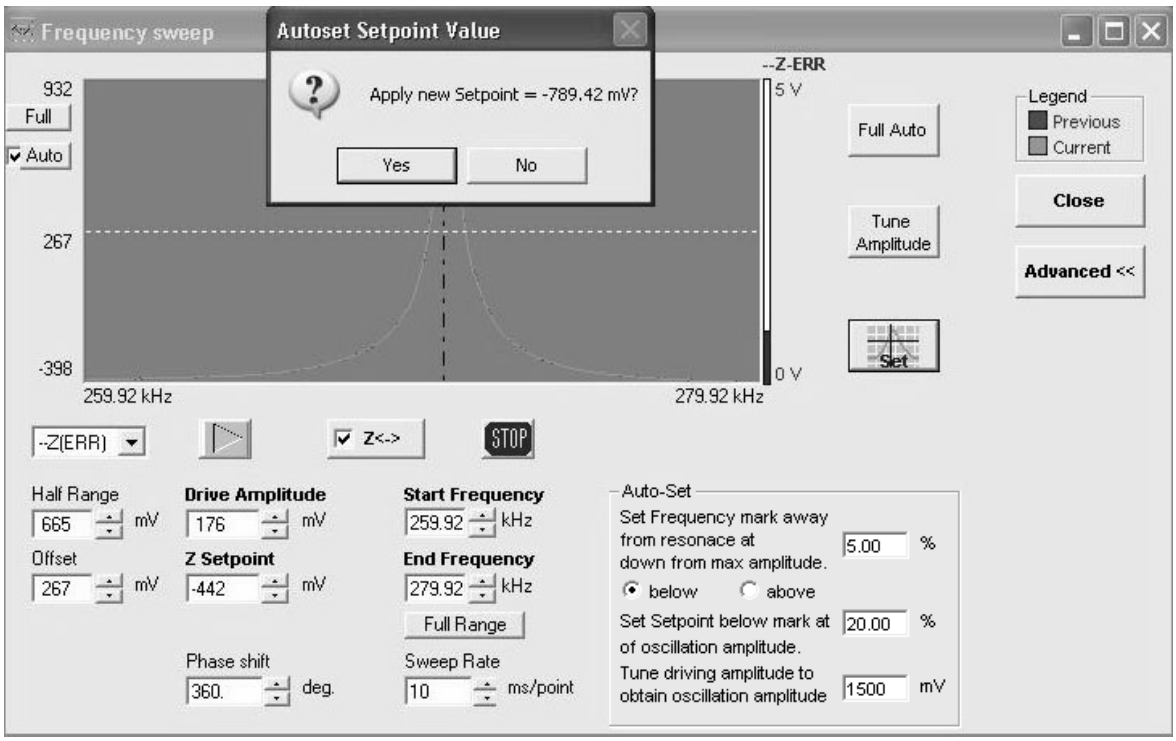


Figure 3.30. Confirm setpoint.

APPROACHING THE SAMPLE

1 Click **Stage** from the EZMode toolbar.

2 Use the focus controls to bring the sample surface into focus on the video monitor.

CAUTION: Whenever you engage the motorized X-Y stage, be sure the probe is a safe distance above the sample/puck.

3 Use the X-Y stage controls to navigate to the largest of the four patterns on the PNI AFM reference (10 µm squares / 20 µm pitch).

Increase or decrease the X-Y step size, as desired, to facilitate both coarse and fine movements.

If necessary, you can orient the sample by simply rotating the puck by hand.

4 Focus on the cantilever.

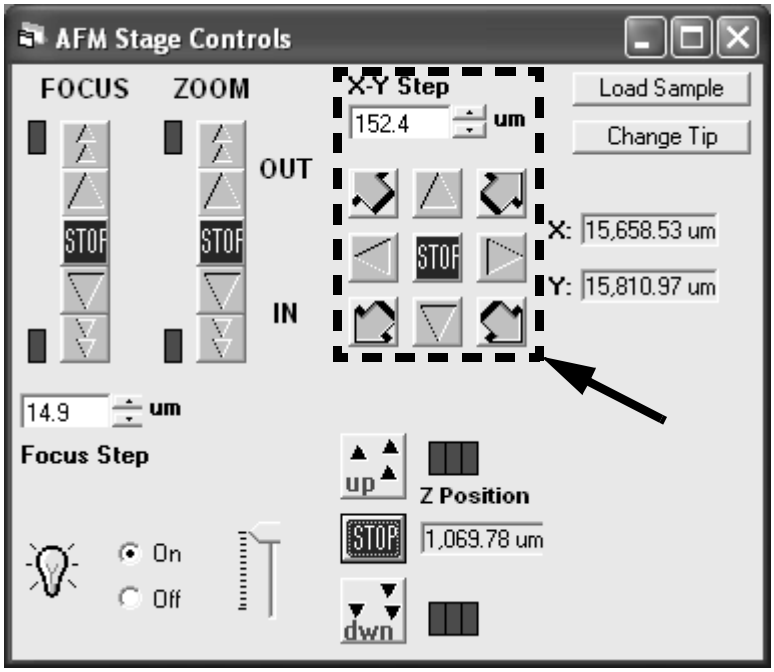


Figure 3.31. X-Y stage controls.

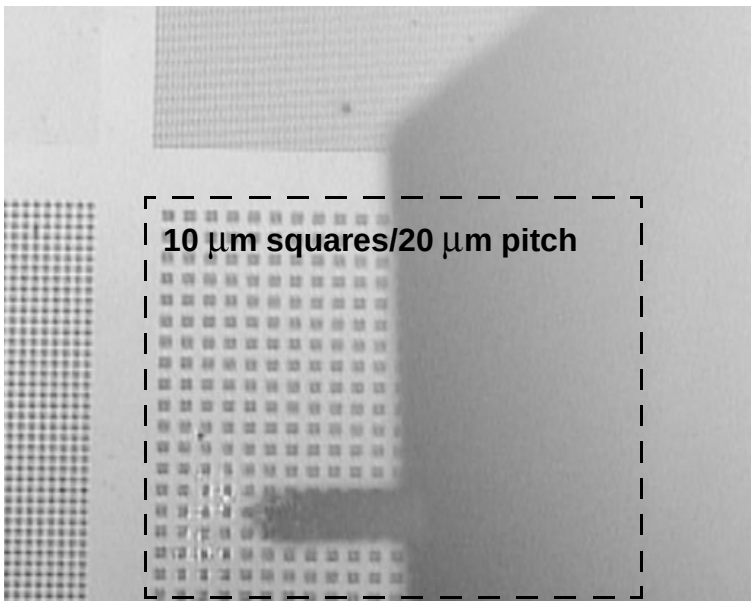


Figure 3.32. Positioning the probe over the scan area.

CAUTION: Be careful not to drive the probe all the way into the sample surface.

5 While carefully monitoring the probe-sample distance by eye, use the 

button to lower the z scanner until the probe is about 1-2 mm above the sample surface.

6 Focus on the sample surface, and make sure the probe is positioned somewhere near the center of the pattern.

7 Click the Tip Approach button on the toolbar.

8 Click OK when the tip approach is complete.

CAUTION: Once the tip approach is complete, and the tip is in contact with the sample surface, do not exit the SPM Cockpit software or turn off the Controller without first retracting the tip. Doing so may cause damage to the tip, scanner, and sample.

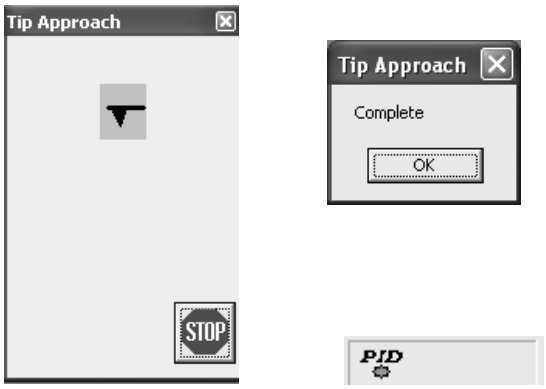


Figure 3.33. Tip approach confirmation.

The PID indicator at the bottom of the window will turn green to indicate that the probe tip is in contact with the sample surface, and the instrument is now ready to perform a scan.

SCAN THE SAMPLE

1 Click the Scan Sample button on the toolbar.

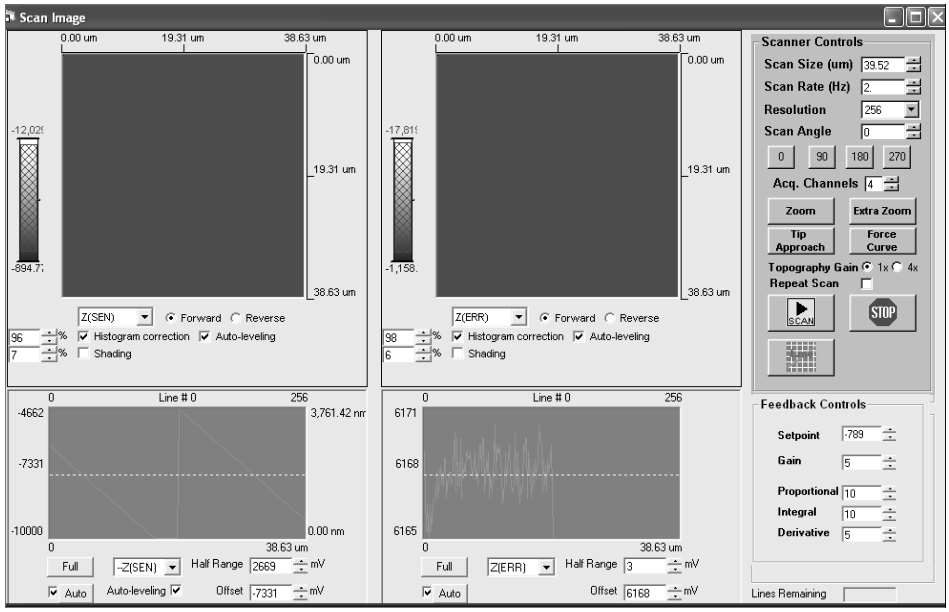
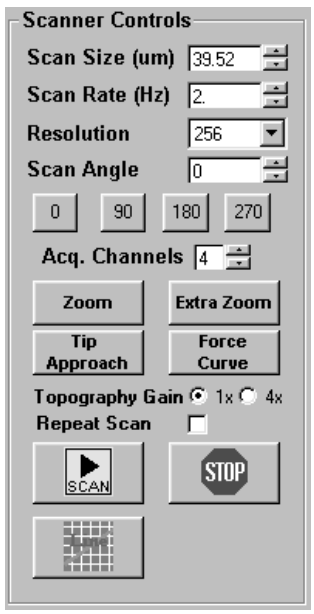


Figure 3.34. Scan image window.

2 Set the scanner controls as follows:

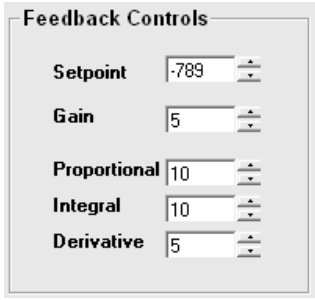
- Scan Size: leave as is*
- Scan Rage: 2 Hz
- Resolution: 256
- Scan Angle: 0
- Acq. Channels: 4
- Topography Gain: 1x

* The default scan size, which is entered by the system when the calibration routine is performed, is the maximum scan area for your scanner.



3 Set the feedback controls as follows:

- Setpoint: leave as is (set automatically when the frequency sweep was performed)
- Gain: 5
- Proportional: 10
- Integral: 10
- Derivative: 5



4 Select the Z(SEN) and Z(ERR) channels from the drop-down menus beneath the two image displays, and for each display, select Forward, Histogram-correction, and Auto-leveling.

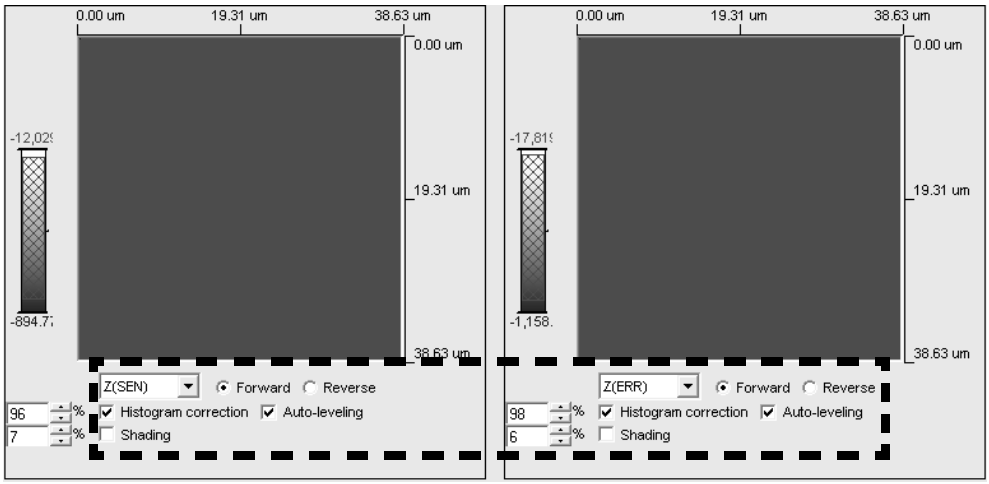


Figure 3.35. Image display settings.

5 Select the Z(SEN) and Z(ERR) channels from the drop-down menus of the two corresponding line scan displays.

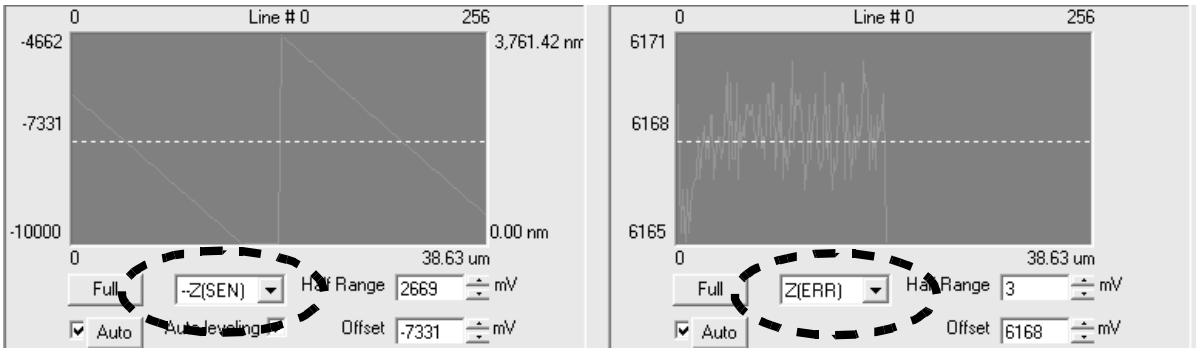


Figure 3.36. Line scan settings.

6 Click  to take a scan.

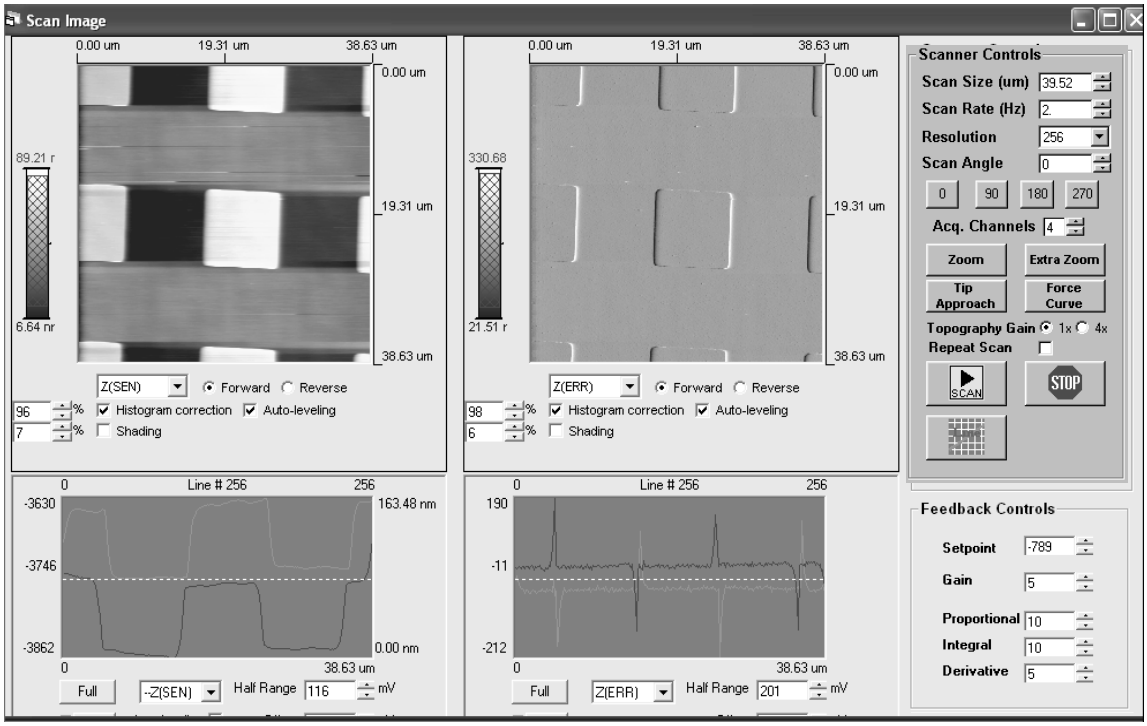
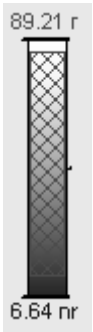


Figure 3.37. Taking a scan.

- The images of the selected channels will build up line-by-line in the displays. If no data is generated, the detector may be out of alignment. In this case, click the STOP button, click Tip Retract from the toolbar, re-align the red dot (page 52), and try another scan.
- To adjust the Z scale of the images, left-click and drag in the bar to the left of each display to select a Z height range.
- To view a single line scan, hold down the SHIFT key and left-click in either image display to define a horizontal line across the image; make sure the line includes the square features. The line scan profile for the Z(SEN) channel should resemble the shape and size of the 10 µm features.



Z scale adjust

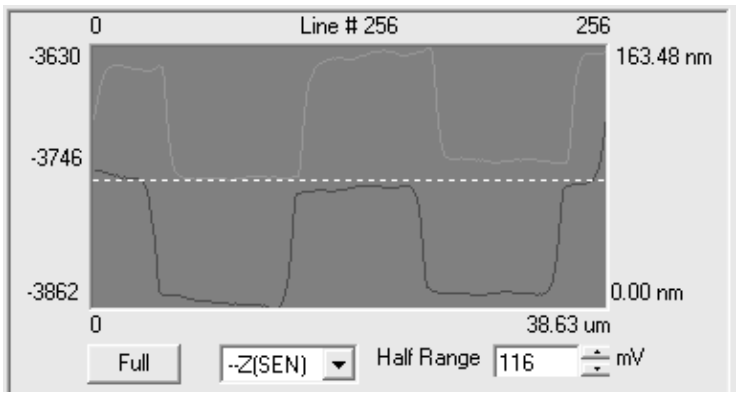


Figure 3.38. Viewing a single line scan: Z(SEN) channel.

When the feedback controls are properly set, the forward and reverse line scan profiles for the Z(ERR) channel will roughly mirror each other.

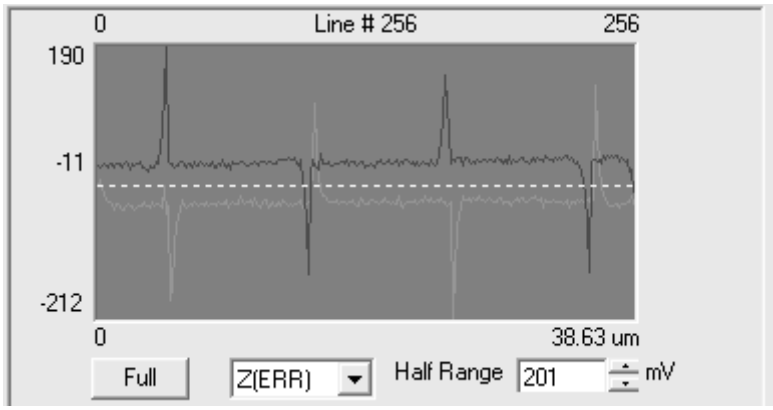


Figure 3.39. Z(ERR) channel line profile.

TROUBLESHOOTING: If you do not see any features, it may be due to one of the following:

- feedback controls improperly set (use the defaults)
- setpoint is too low (increase slowly, a maximum of 2-4 clicks at a time, and watch for a response in the display—increasing too much can break the tip or damage your sample)
- resonant frequency not properly set (open the Frequency sweep window, and perform the frequency sweep procedure again)
- dull probe tip (replace tip)
- broken tip (replace tip)
- poor mechanical coupling between the probe and scanner (reseat probe)

7 To take additional scans, click  again, or check Repeat Scan to take continuous scans of the same region.

8 To zoom to a new scan region:

a) Left-click and drag in the image display to define a scan area.

b) Left-click again to position the box within the scan region.

c) Right-click to confirm the new scan region.

The probe will move to the new scan region, where you can start a new scan. Additional zoom features are accessible via the Zoom and Extra Zoom buttons, or by simply double-clicking in the display.

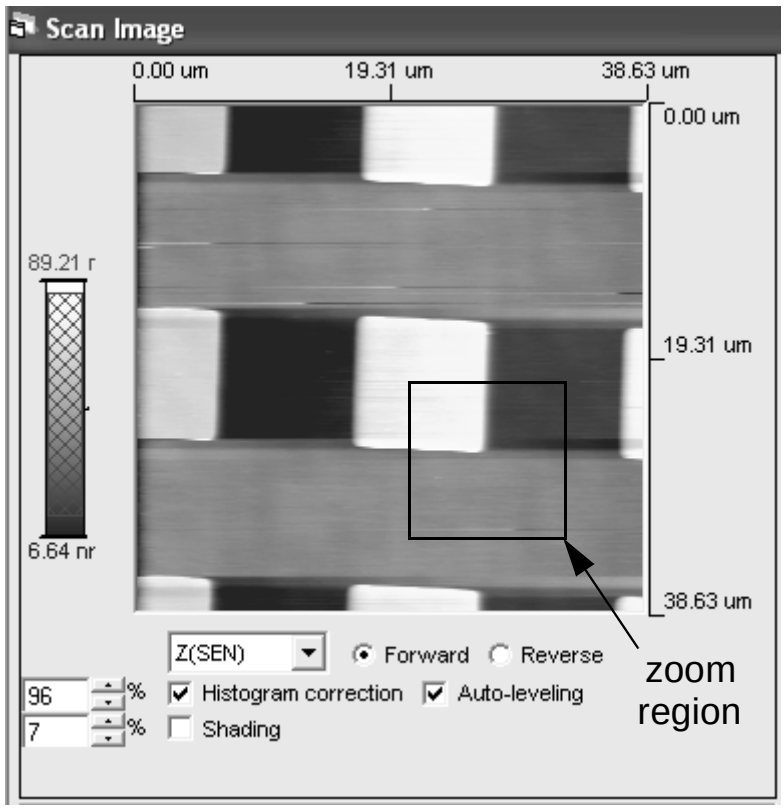


Figure 3.40. Selecting a zoomed-in scan region.

9 To end your session now, click Tip Retract on the EZMode toolbar.

Once the tip is retracted, it is safe to turn off the Master Computer and the Controller.

IMAGE PROCESSING

1 Click Image Processing on the EZMode tool bar.

2 Click OK to load the Z(SEN) image into the image processing display.

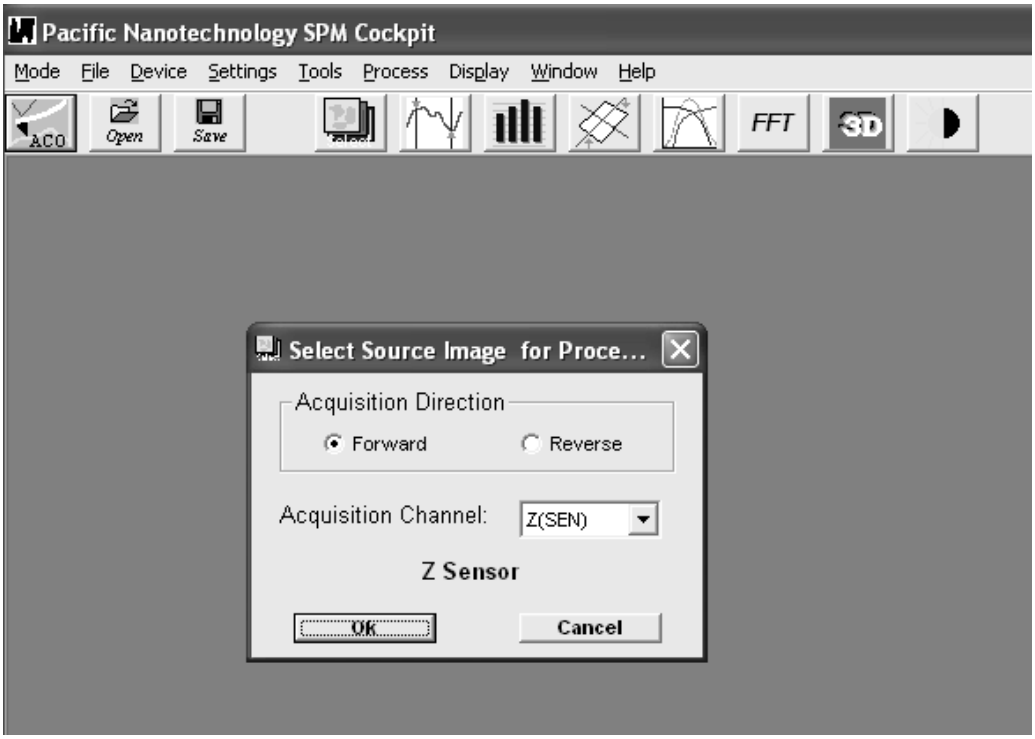


Figure 3.41. Image processing module.

3 Select the desired acquisition channel and direction for the image to be processed.

The raw image data will not closely resemble the image in the scan image window, as some basic real-time image processing was applied as it was being acquired.

4 Click  to apply a plane correction.

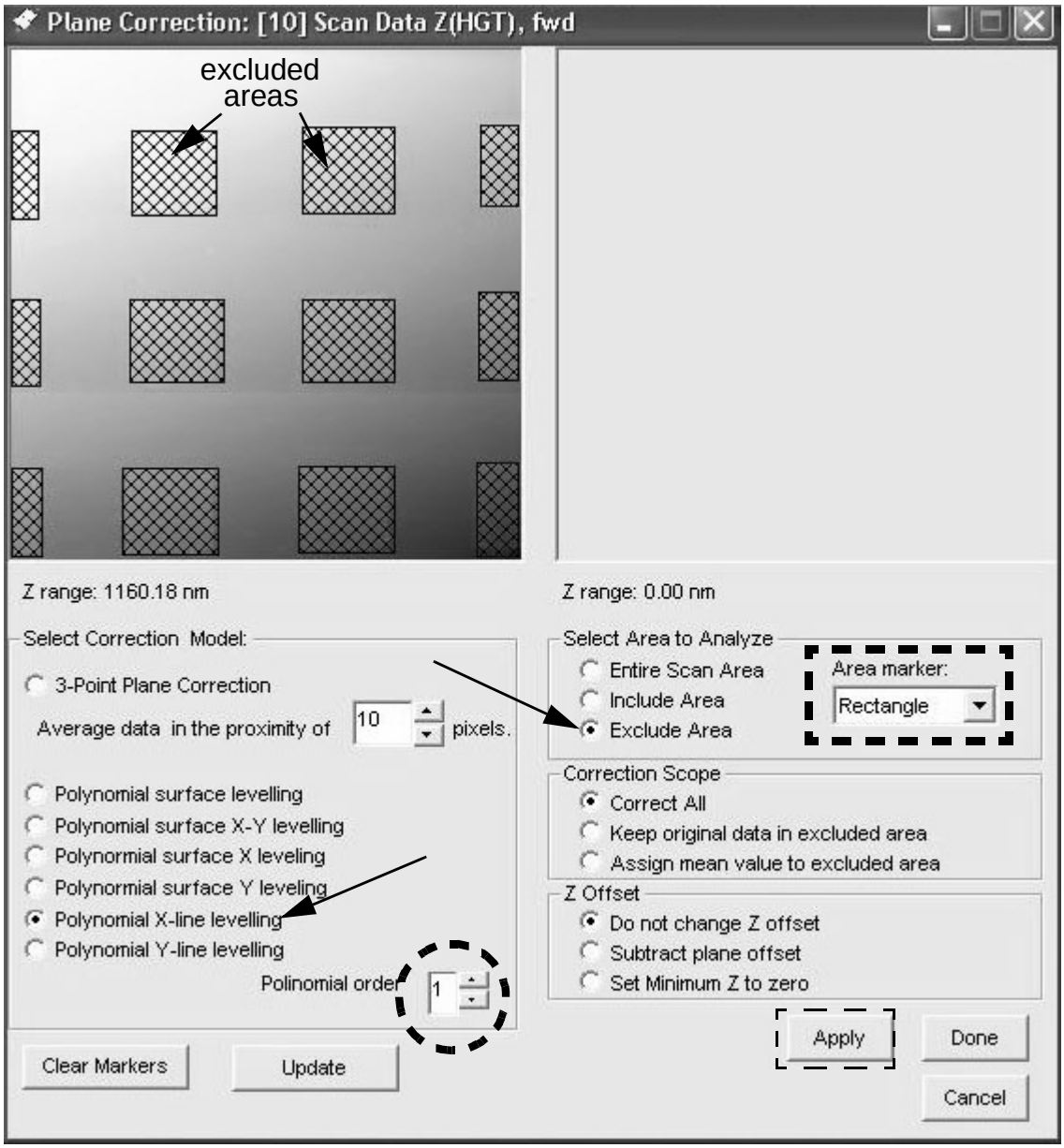


Figure 3.42. Plane leveling tool.

a) Under Select Correction Model, select:
- Polynomial X-line leveling
- Polynomial order: 1

b) Under Select Area to Analyze, select:
- Exclude Area
- Area marker: Rectangle.

c) To exclude the features on the PNI AFM reference, use the mouse to left-click and drag in the image display so that every feature (both whole and partial) is completely covered.

d) Click Apply, and the leveled image appears in the right-hand display.

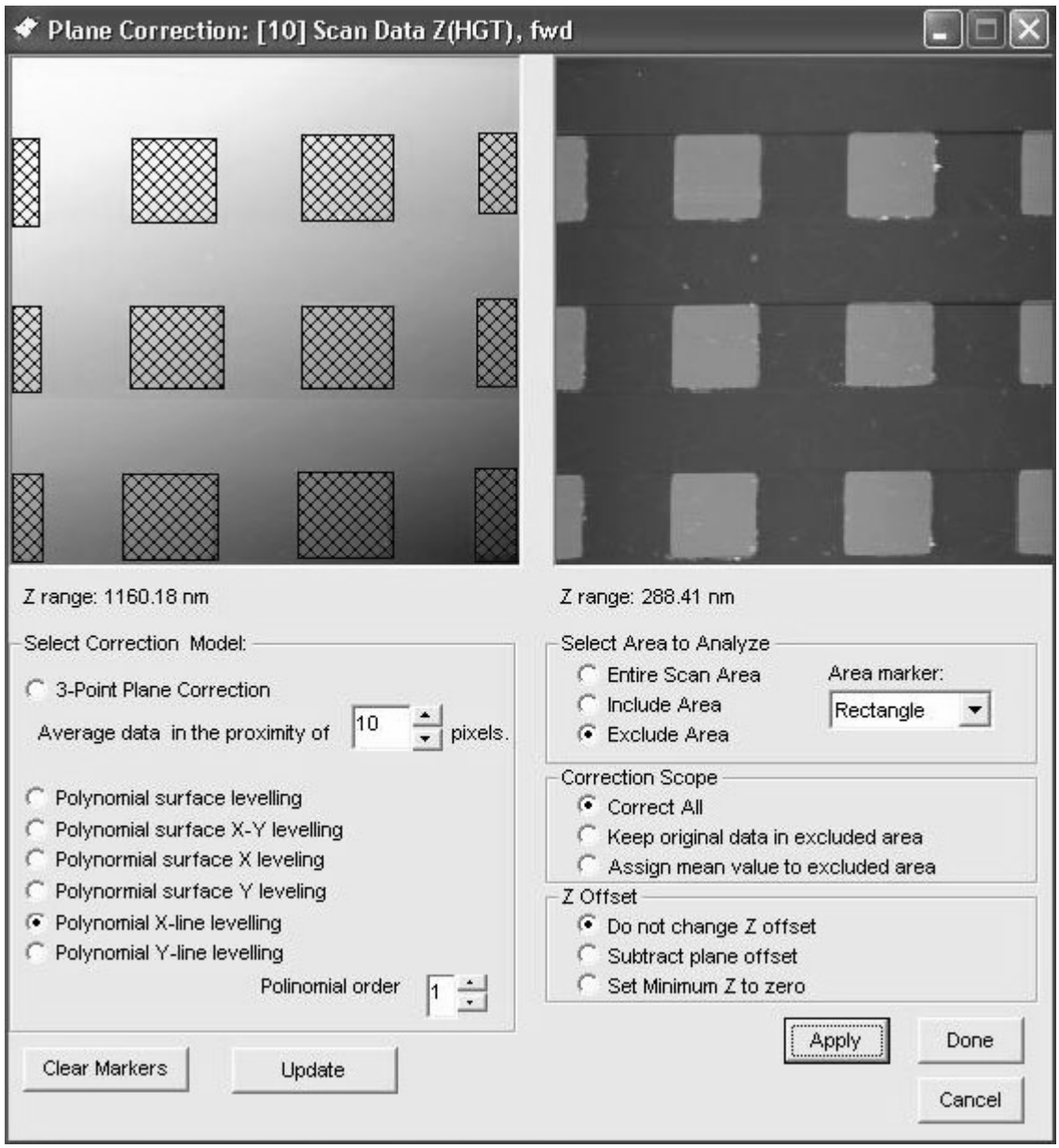


Figure 3.43. Leveled image (right).

5 Click  to open the line profile tool.

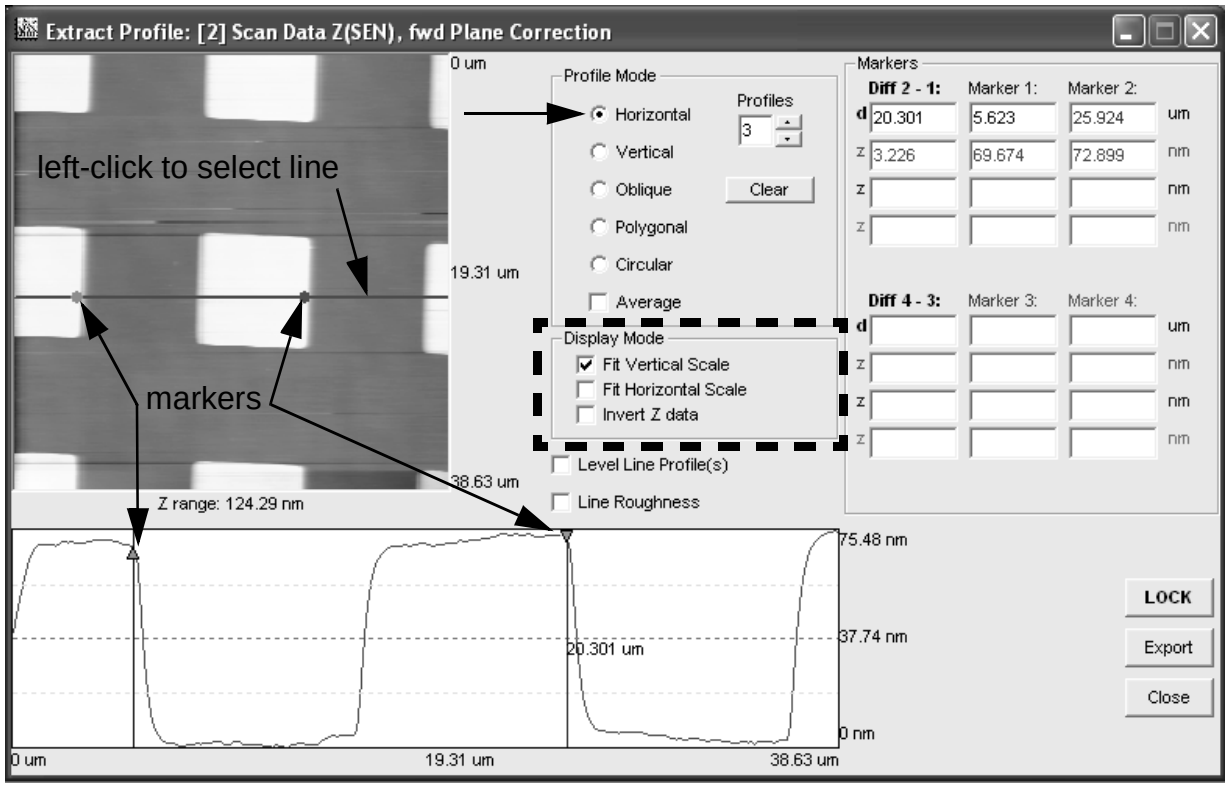


Figure 3.44. Line profile tool.

a) Under Profile Mode, select Horizontal.

b) Under Display Mode:

- Check Fit Vertical Scale
- Uncheck Invert Data

c) Left-click in the image display to select a line.

d) Left-click in the line display to make measurement markers.

In the example above, measurements are made between the edges of two consecutive features on the PNI AFM reference. The measurements displayed to the right confirm a pitch of 20 µm and a Z-height of approximately 70 nm.

NOTE: These measurements should not be used to calibrate your instrument!

6 Click  to open the histogram tool, and use the slider bars to mark the middle of the two ranges where the z data points are clustered.

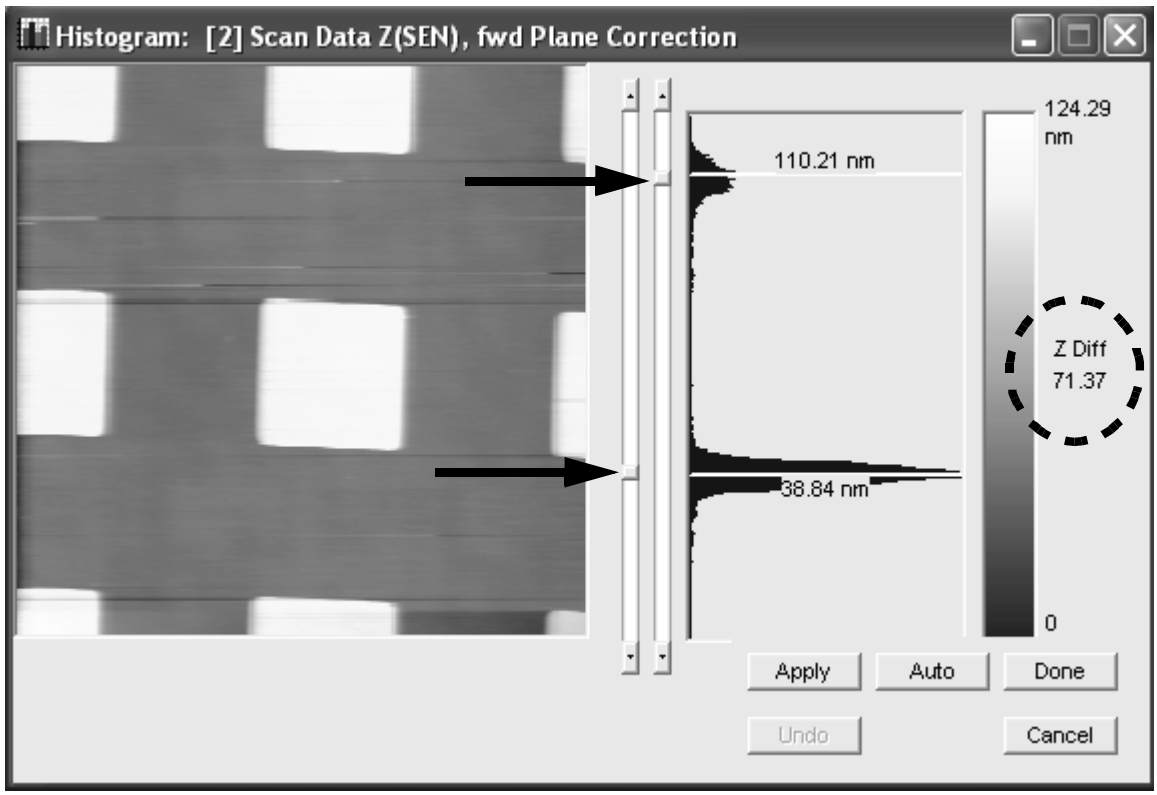


Figure 3.45. Histogram tool.

The Z Diff measurement on the vertical bar confirms the 70 nm height of the PNI AFM reference features.

7 To save any of your processed images, select File→Save Image(s).

8 Click  to return to the acquisition module.

CAUTION: To prevent damage to your scanner, probe, and sample, be sure to retract the tip before exiting the SPM Cockpit software or turning off the Controller.

9 Click Tip Retract.

It is now safe to exit the SPM Cockpit software.

Chapter 4

Material Sensing Modes

INTRODUCTION

The Nano-R AFM is capable of providing much more than topographical information about your sample. By monitoring other signal channels which are available when taking an AFM image, information about the properties of your sample surface can be obtained.

Material sensing modes include, but are not limited to, lateral force microscopy (LFM), phase imaging, and force vs. distance curves. Refer to the AFM Tutorial (page xxix) for more information.

WARNING: *Before operating the Nano-R AFM, make sure you are familiar with the safety information on page vi.*

LATERAL FORCE MICROSCOPY (LFM)

LFM studies are done while operating in contact mode. The Z(L-R) channel, one of the four channels available in contact mode, provides lateral force information. The resulting LFM image can then be compared to the images generated by the other channels.

CAUTION: To prevent damage to your instrument, probe, and sample, make sure you are familiar with the caution statements in Chapter 2.

1. Set up the Nano-R to take an image in contact mode, as described in Chapter 2.

2. Set the scanner and feedback controls as described on page 29.

3 Select the Z(SEN) and Z(L-R) channels from the drop-down menus beneath the two image displays, and for each display, select Forward, Histogram-correction, and Auto-leveling.

4 Select Settings→Input Selects to ADC.

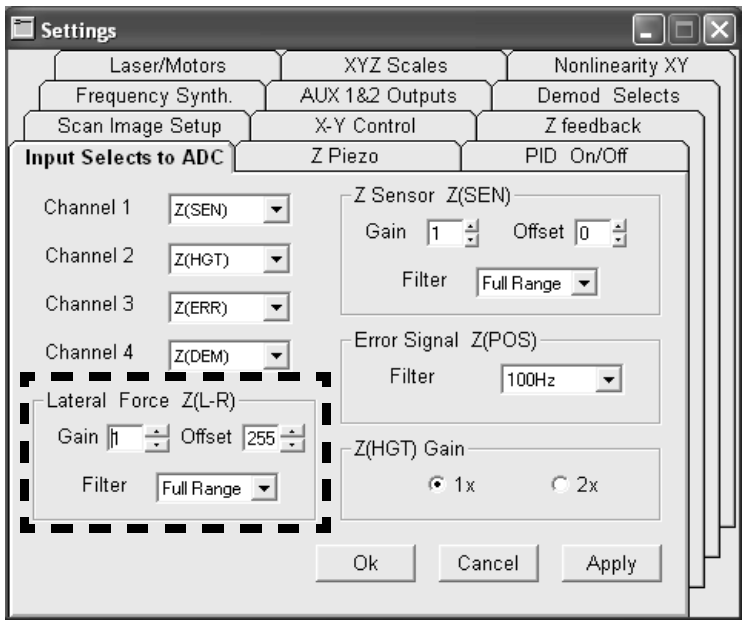
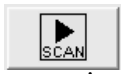


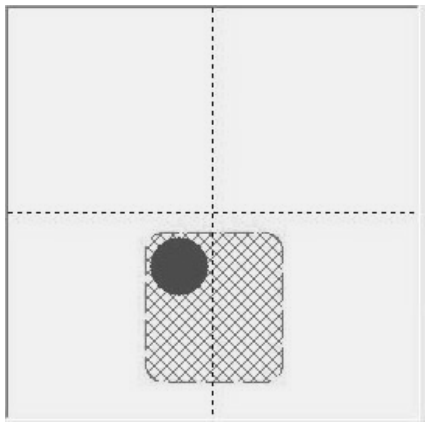
Figure 4.1. LFM settings.

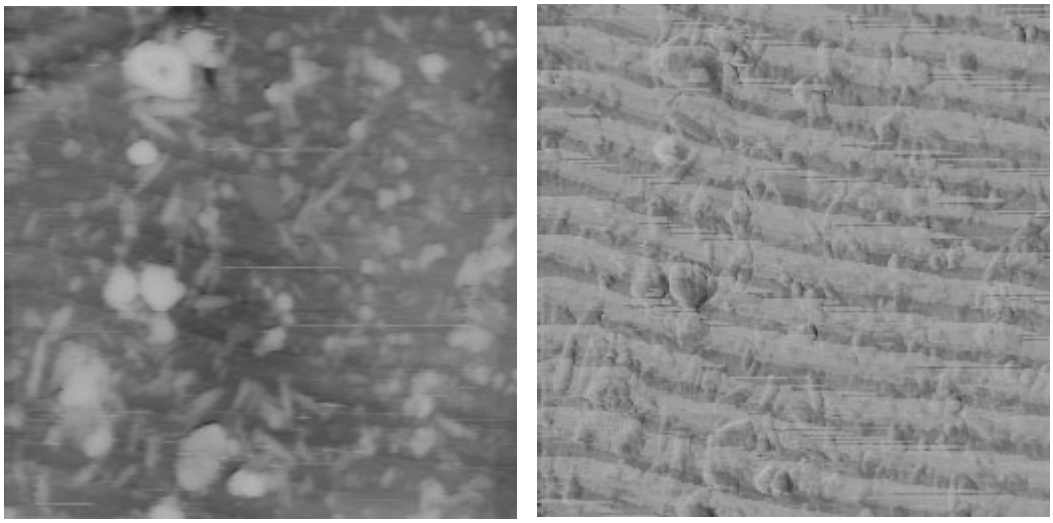
5 For Lateral Force Z(L-R), set the Gain to 1 and the Offset to 255.

6 Click 

to take a scan.
While scanning, you can monitor the image and line scan of any of the four channels.

Normally, the gain and offset values for the Z(L-R) channel should be sufficient for most lateral force imaging situations. If a higher gain is needed, open the Red Dot Alignment window and align the photodetector so that the red dot is to the left of the vertical mid-line, near the left border of the green zone. The gain, offset, and filter can then be adjusted while scanning, for optimal image acquisition.





Topography **LFM**

Figure 4.2. 6x6 µm image of composite material.

PHASE IMAGING

Phase imaging is done in close-contact mode. The Z(DEM) channel, one of the four channels available in close-contact mode, provides phase information. This channel can be set to represent changes in either the phase or the amplitude of the cantilever vibration, while holding the other constant.

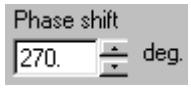
CAUTION: To prevent damage to your instrument, probe, and sample, make sure you are familiar with the caution statements in Chapter 3.

SET UP

Set up the Nano-R to take an image in close-contact mode, as described in Chapter 3.

FREQUENCY SWEEP

When setting the resonant frequency (in the Frequency Sweep window), first set the Phase Shift to 270.



SCANNING

1 Set the scanner and feedback controls as described on page 29.

2 Select the Z(SEN) and Z(DEM) channels from the drop-down menus beneath the two image displays, and for each display, select Forward, Histogram-correction, and Auto-leveling.

3 Select Settings→Demod Selects.

4 Set Demodulation to Amplitude.

When set to Amplitude (the default setting), the Z(DEM) channel represents changes in the phase while a constant amplitude is maintained.

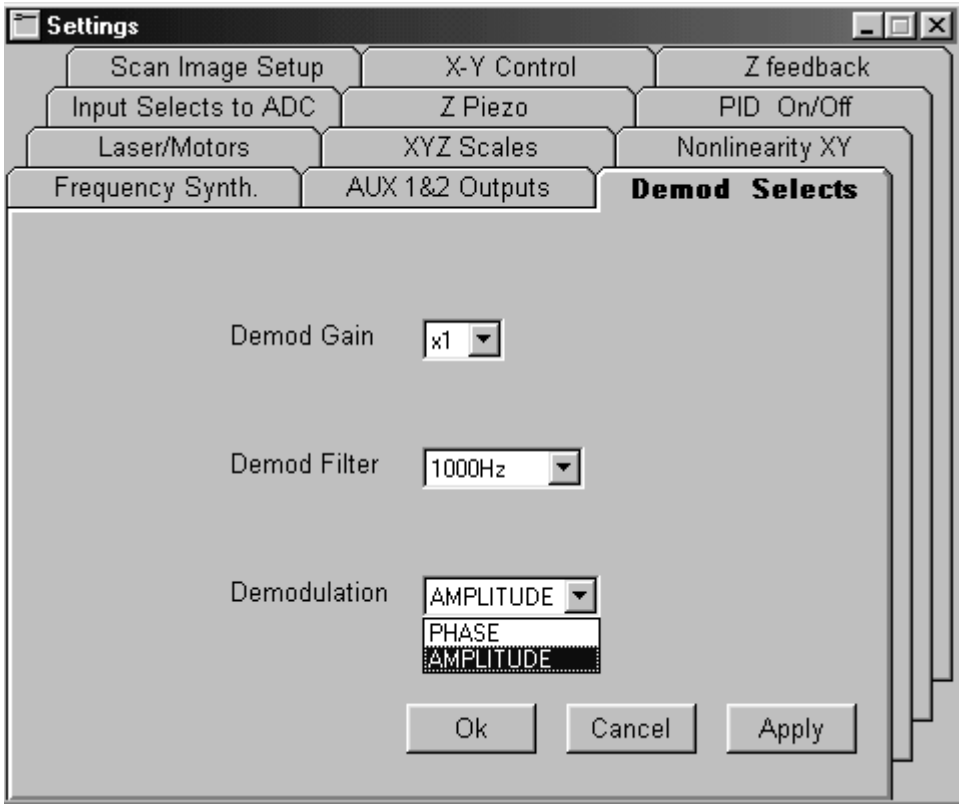
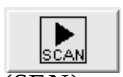


Figure 4.3. Z(DEM) channel settings.

5 Click 

to take a scan.

The Z(SEN) and Z(DEM) channels were used to generate the images of the PNI AFM reference shown below. While the surface of the PNI AFM reference is composed of a nominally homogeneous material, the phase image (right) reveals surface contaminants.

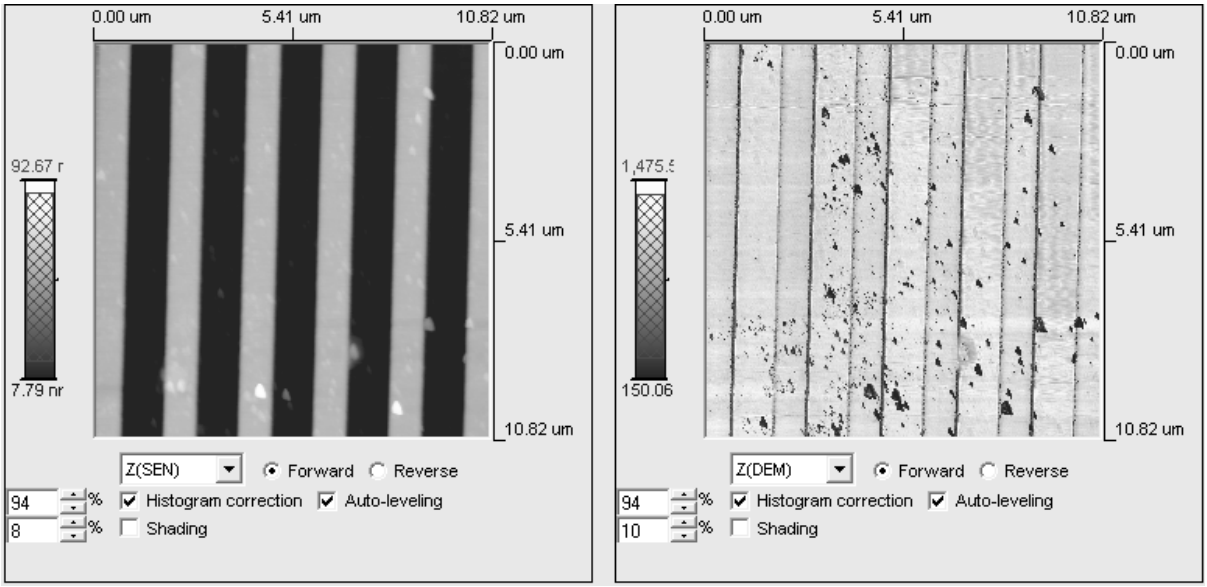
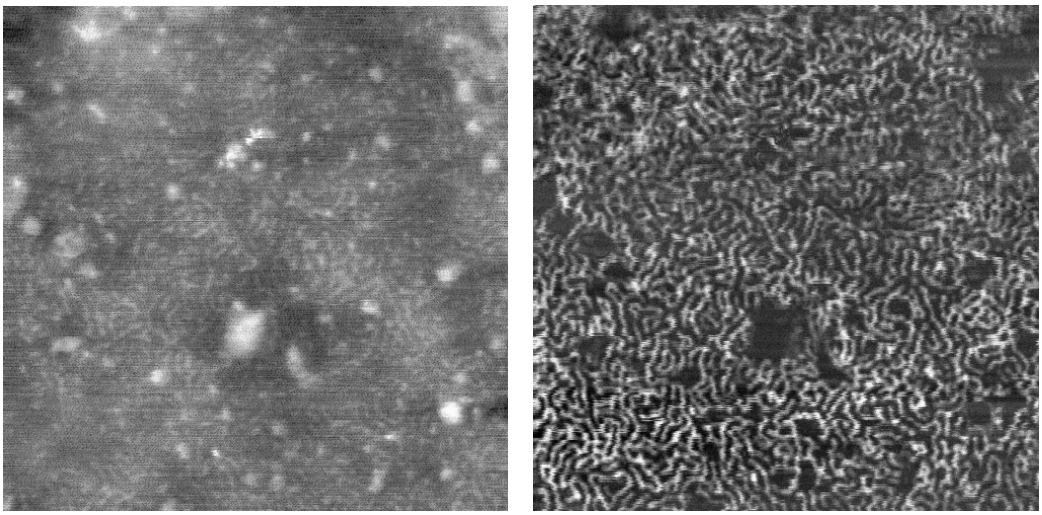


Figure 4.4. PNI AFM reference: Topography (left) and Phase (right).

The following images of a sample of SBS film compare the topography (Z(SEN) channel) information with the phase (Z(DEM) channel) information.



Topography **Phase**

Figure 4.5. 1.5x1.5 µm image of SBS film.

Note that phase information is a convolution of several factors, with contributions from material properties as well as topography. Phase imaging typically enhances fine feature contrast and provides some qualitative information about visco-elasticity, hardness, adhesion, and contaminations.

FORCE-DISTANCE CURVES

Single-point measurements can be taken at selected locations on your sample surface. The probe is moved toward the sample surface, to a pre-set voltage-defined position, and then retracted. The amount of cantilever deflection over the course of this movement is expressed by the Z(ERR) signal, which is used to generate a curve.

CAUTION: To prevent damage to your instrument, probe, and sample, make sure you are familiar with the caution statements in Chapter 2.

1 Take an image of your sample in contact mode, as described in Chapter 2.

2 To select a point on the sample to take a measurement, hold down the CTRL key and left-click in the image display (in the Scan Image window). A black dot marks the location.

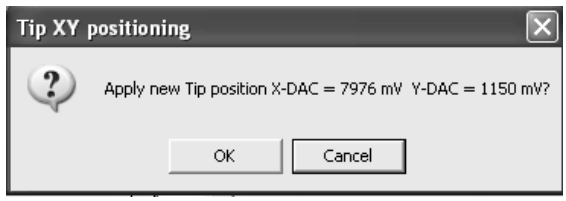


Figure 4.6. Selecting a measurement location.

When you click OK, the tip will move to the selected location.

3 Click Force Curve to open the Force / Distance Curve window.

4 Check the Auto option.

5 Make sure Z(ERR) is the selected signal from the signal drop-down menu.

6 Make sure the correct spring constant value for the cantilever you are using is entered in the Spring Constant field.

For PNI pre-mounted contact probes, this value is 0.2 N / m. When entering a new value, you must click >> to apply it.

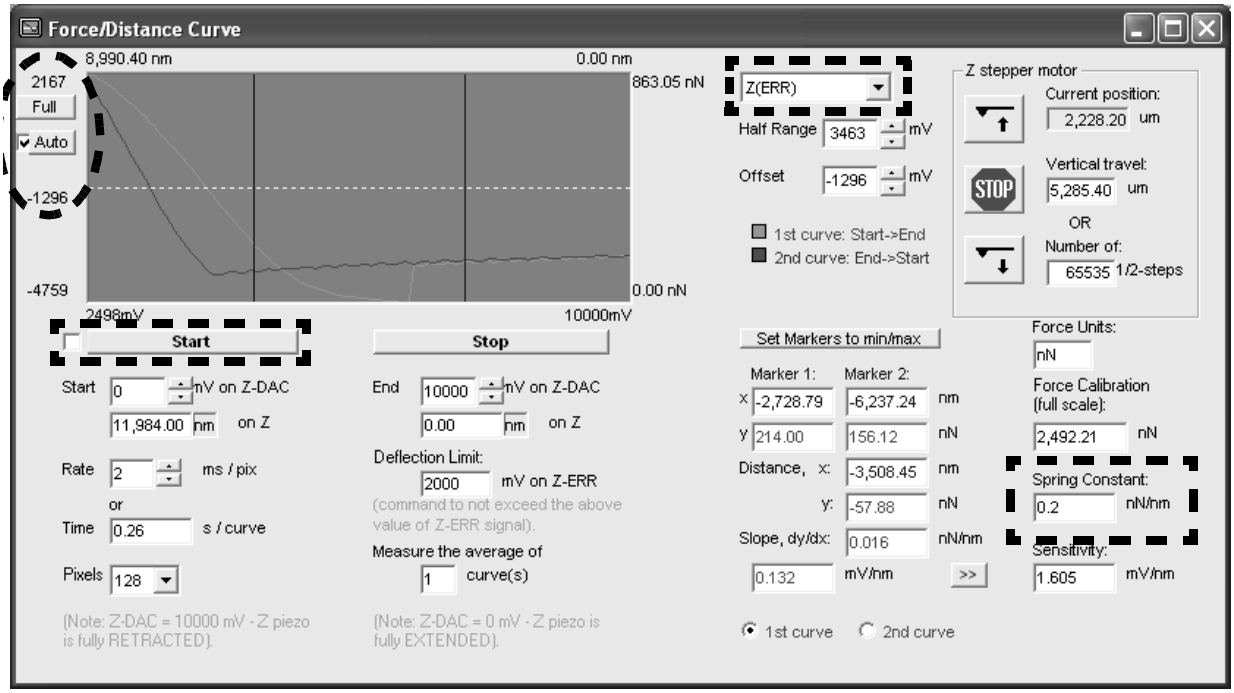


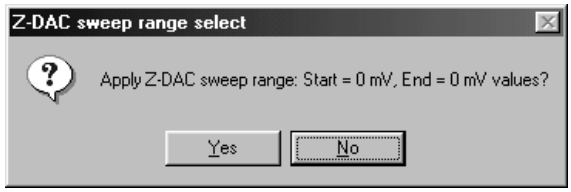
Figure 4.7. Force-distance curve settings.

7 Click Start to generate a force-distance curve.

The first curve (displayed in green) represents the deflection of the cantilever as it approaches the sample surface. The second curve (displayed in red) represents the retraction of the cantilever.

8 Zoom in on a range of interest by left-clicking on the curve and dragging to define the zoom region.

9 Right-click to define the second limit of the range, and then click Yes to apply it.



Confirm sweep range

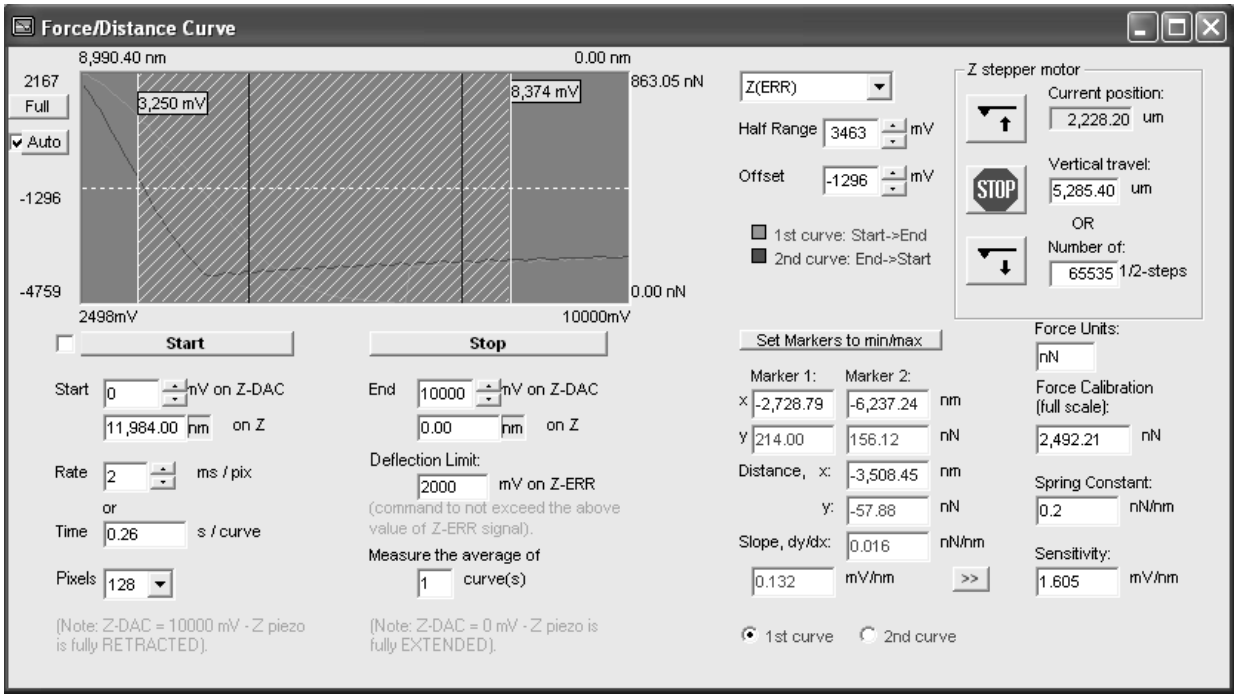


Figure 4.8. Zooming in on a voltage sweep range.

The force-distance curve window will re-scale to the new voltage range for subsequent curves.

Measurements can be taken as follows:

- Left-click anywhere on the curve to measure Z height.
- Right-click anywhere on the curve to measure the signal level.
- Left-click to grab and move the two measurement markers.
- To take continuous measurements, check the box next to the Start button.

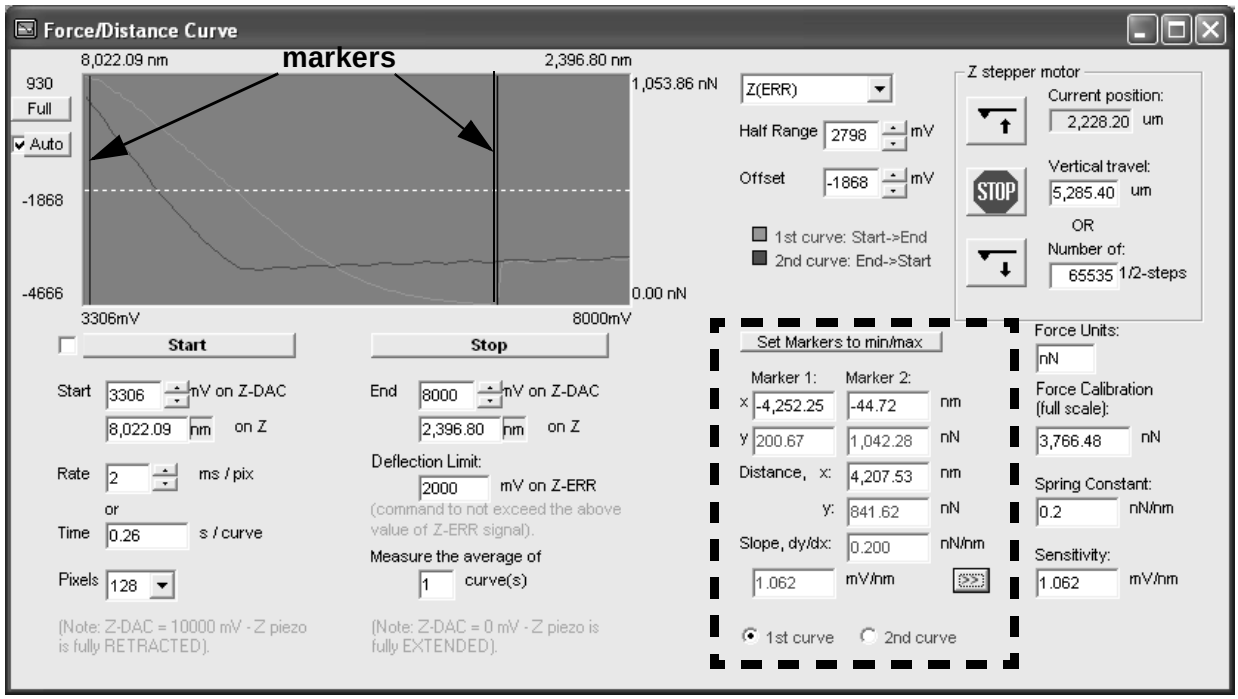


Figure 4.9. Taking measurements.

Chapter 5

X'Pert Mode & More

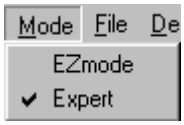
INTRODUCTION

The tutorials in Chapter 2 and Chapter 3 guide you through the minimal steps required to take an AFM image. This chapter takes you a little further, exploring some of the Nano-R features and functions that can help you take better images. The contents are organized functionally, roughly following the basic steps for taking an image outlined on page 10.

WARNING: *Before operating the Nano-R AFM, make sure you are familiar with the safety information on page vi.*

CAUTION: To prevent damage to your scanner, probe, and sample, make sure you are familiar with the caution statements in Chapter 2 and Chapter 3.

X'PERT MODE



Once you are comfortable taking images in EZMode, you may find it more convenient to operate in X'Pert Mode. Select Mode→Expert to display the X'Pert Mode short-cut buttons, which provide access to all the steps required for taking an AFM image, as well as other functions and tools.

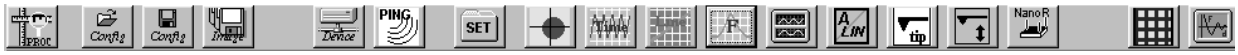


Figure 5.1. X'Pert Mode short-cut buttons.

Display the image processing toolbar.

Open the configuration file to be used for this session.

Save the current parameters and settings as a new configuration file.

Save image file.

Select the device directory.

Test the connection with the Controller.

Display the tabs for all the Settings menu items.

Open the Red Dot Alignment window.

Time mode oscilloscope.

Line mode oscilloscope.

Open Frequency sweep window.

Dual-trace storage scope.

Perform X-Y scanner calibration routine.

Automatic tip approach and retract.

Manual tip up / down control, with signal monitoring.

Advanced AFM stage controls.

Open Scan image window.

Open Force-distance curve window.

CONFIG FILES

Two configuration files are supplied with the Nano-R, one for contact operation and one for close-contact. These files contain information that is unique to your particular instrument. Therefore, it is very important that back-up copies of these files be kept in a safe place in the event that the ones on your Master Computer are accidentally altered or deleted.

These files also contain the factory default values for all the software settings that control your instrument.

OPENING

At the start of each session, you need to load a configuration file. The filenames for the two supplied configuration files are in the following format (xxxx is the serial number of your Nano-R instrument):

- for contact mode: sxxxx-con.cfg
- for close-contact mode: sxxxx-osc.cfg

You can use one of the supplied files or a user-created file containing the settings from a previous session. However, the type of configuration file—contact or close-contact—must match the imaging mode for your session (see below). Once you have loaded a configuration file in X'Pert mode, there is no need to also select the imaging mode, as in EZMode.

SAVING

In the course of taking images, you will invariably change many settings and parameters. At any point, the current settings, which may apply to a particular sample and / or application, can be conveniently saved for future use by saving them in a new configuration file.

When saving new configuration files, the filename should identify the file as either contact or close-contact. If you load a contact configuration file and attempt to operate in close-contact mode using a close-contact probe, for example, you will not be able to do so.

STAGE CONTROLS

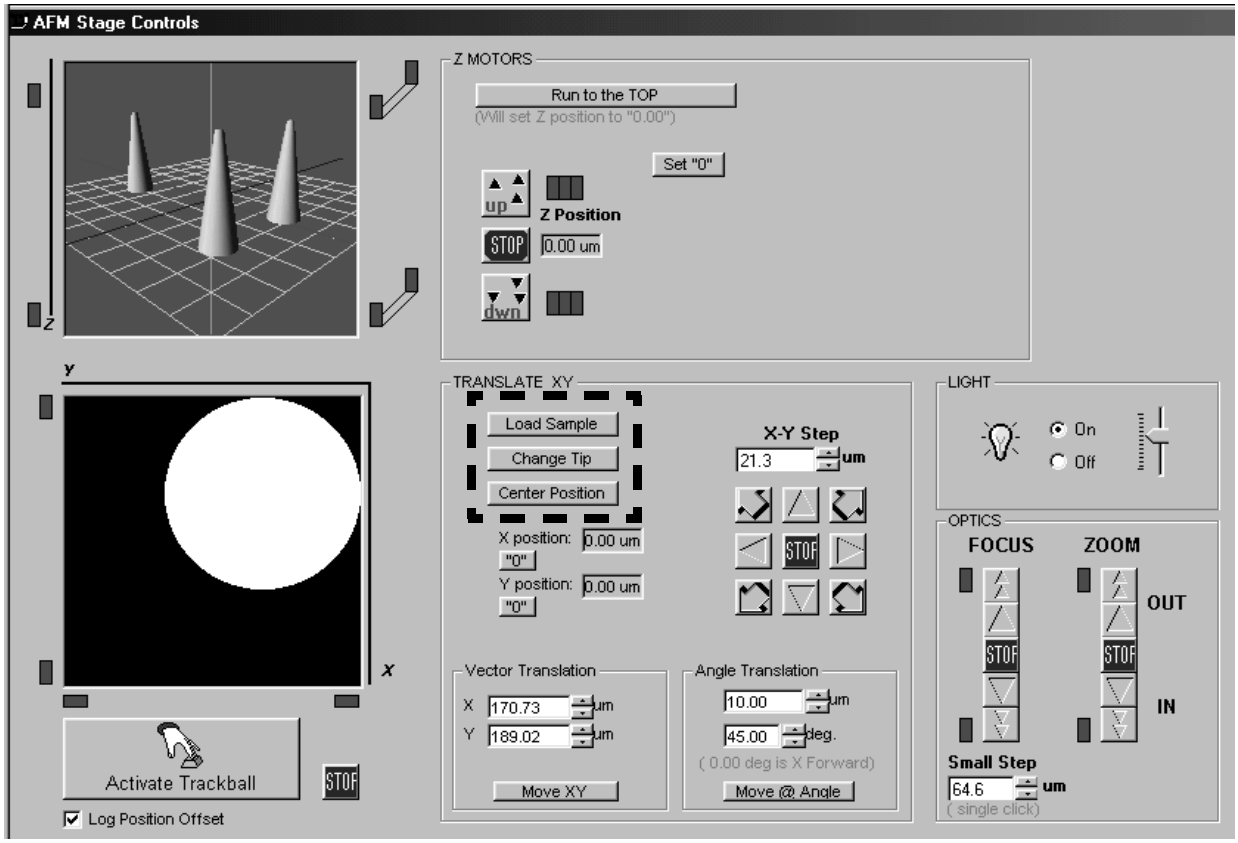


Figure 5.2. Advanced stage controls window.

Click  to access the advanced stage controls. Buttons in the Translate XY box provide quick, automated ways to perform the stage translations for changing the probe and sample.

The Load Sample button automatically moves the puck to the limit of the X-Y stage range to facilitate changing the sample. Once a sample has been mounted and the puck replaced, you can click the Center Position button to return the puck to its original position.

The Change Tip button will run the Z motors to the top of their range (both the scanner head and the video objective), to facilitate installing a new probe. The Run to the TOP button in the Z Motors box does the same thing.

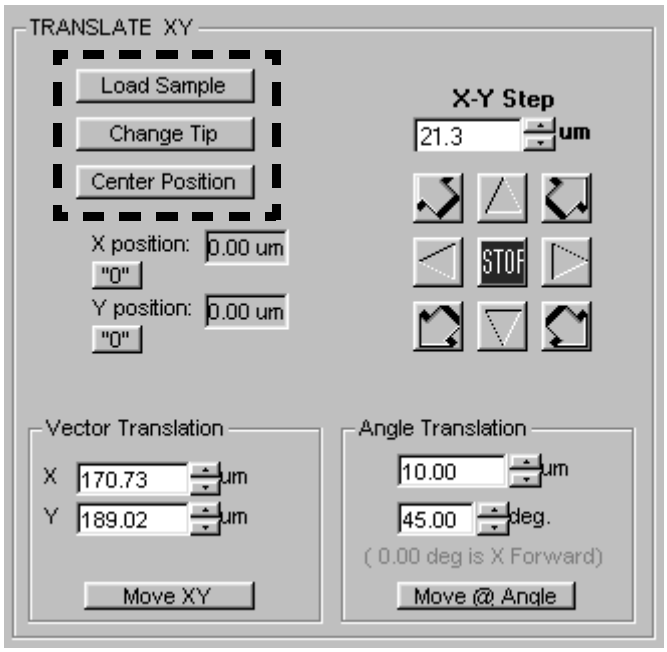


Figure 5.3. X-Y Stage controls.

Trackball

A trackball is supplied with the Nano-R system as an alternative way of accessing the motorized stage controls. The trackball can be activated from the stage controls window in either EZMode (Stage) or X'Pert Mode (NanoR or Tools→NanoR Stage).

Trackball Stage Controls

	Left button	Right button
X-Y Stage	off	off
Z Stage	off	on
Zoom	on	on
Focus	on	off

SAMPLE MOUNTING

The sample should be mounted so that it is stable and relatively flat. The magnet at the center of the puck is a convenient way to stabilize the sample. Double-sided tape is another method.

The height of the sample puck can be adjusted to accommodate samples of varying heights. The puck is composed of 5 layers each measuring 1/4" in height. Therefore, if your sample is taller than 1/4", you should remove one layer for each 1/4" of height in your sample.

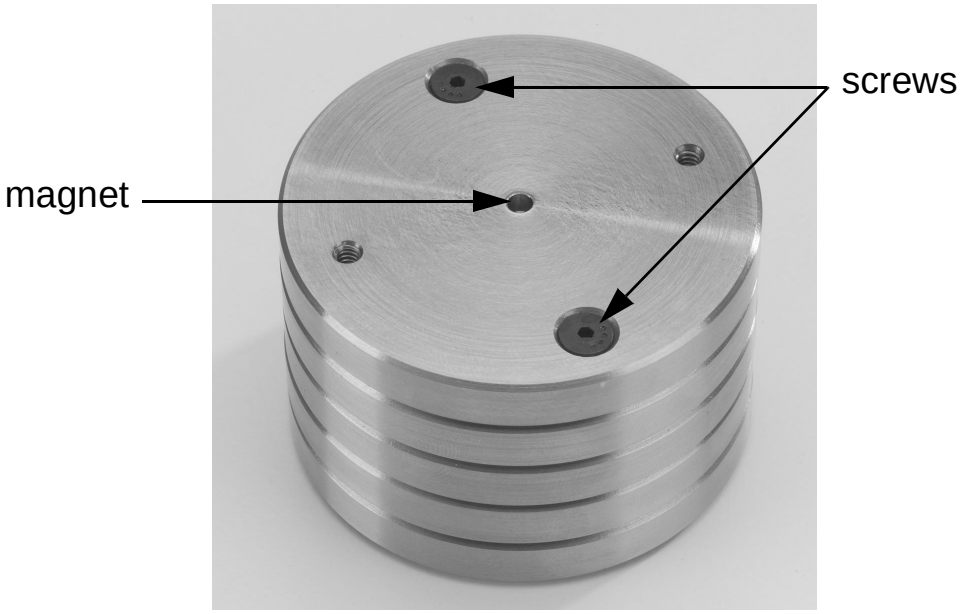


Figure 5.4. Sample puck.

Use a 1/16" Allen wrench to loosen one of the screws on the top of the puck. Loosen it only until you feel some resistance, then loosen the other screw completely. Finally, finish loosening the first screw and remove the puck layer. To add a layer, tighten the screws in the same way.

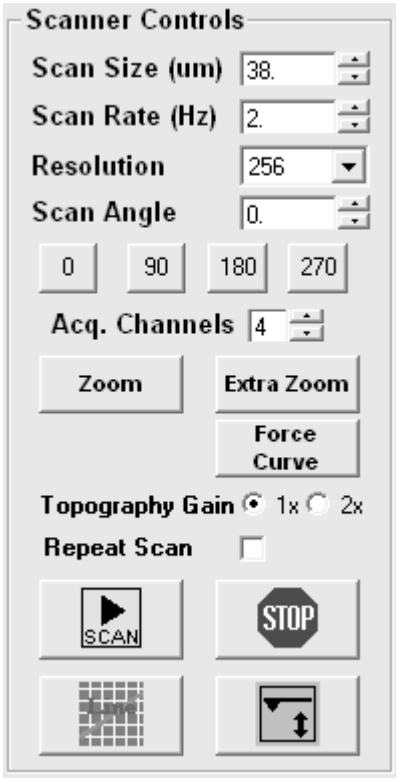
SCANNING

The  button on the X'Pert Mode toolbar opens the Scan image window. While this is the same window used in EZMode, this section provides additional details about the meaning of the various settings.

Scan Size

The maximum scan area that your instrument's scanner can accurately scan is automatically entered in the Scan Size field each time the calibration routine is performed.

Scan Rate



As a general rule, the slower the scan rate, the better the feedback loop is able to track the sample topography. Therefore, the scan rate will largely depend on how rough or smooth the sample is. For example, if the sample is very flat, scanning at a slow rate is of no benefit; and if a rough sample is scanned too quickly, information is likely to be lost.

Resolution

This value represents the number of pixels per line in the image. The default setting, 256, will result in a 256x256 pixel image (i.e., 256 line scans, each consisting of 256 data points).

Scan Angle

The scan region is a square area that can be rotated as desired, rather than having to physically rotate the sample. Note that the scan size may be automatically reduced in the event that the rotation causes some of the scan area to extend beyond the range of your scanner's maximum range.

Zoom

The zoom windows provide tools for defining and fine-tuning your scan area. These windows can also be accessed by double-clicking in the image display. Simple zooms can be accomplished by clicking and dragging in the image display to define a new scan area.

Topography Gain

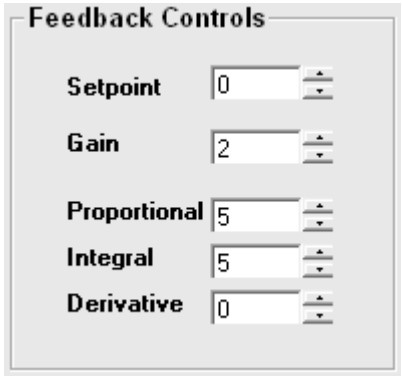
Increasing the topography gain to 2x may be useful when imaging very small features (< 5 nm). This is a way of increasing the gain without losing resolution. The Z(HGT) channel should be monitored in this situation (instead of Z(SEN)), as the z sensor will not be sensitive enough to resolve the small features.

Repeat Scan

When this option is checked, the system will take a continuous scan of the same region. This allows you to keep adjusting the scanner and feedback controls until they are optimized.

FEEDBACK CONTROLS

When you begin scanning, use the default feedback control settings. These can then be adjusted while scanning to optimize image acquisition. The parameters should be adjusted one at a time, in small increments. Allow the system to scan a few lines after each adjustment so you can see the result before adjusting further.



Adjust these settings carefully, as it is possible to damage your scanner, tip, and sample.

Setpoint

The setpoint represents the tip-sample distance that the feedback electronics maintains as the tip is scanned over the sample surface. In contact mode, this is expressed as a force (in nanonewtons); raising the value brings the tip closer to the sample. In close-contact mode, the setpoint is expressed as a voltage, which is related to the voltage required to oscillate the cantilever at (or near) its resonant frequency. The setpoint in close-contact mode is set automatically when the frequency sweep is performed. Increasing this value (making it less negative) brings the tip closer to the sample.

Gain

The gain should be adjusted 1-2 steps at a time. Increasing the gain too quickly can result in damage to the scanner.

PID

Use the following guidelines for tuning the proportional, integral, and derivative gains (PID):

- In contact mode, the derivative should be kept at 0, otherwise the scan will be unstable.

- For relatively flat samples, use a relatively high proportional gain while keeping the derivative low.
- For relatively rough samples, use lower proportional values while increasing the integral.

SAVING IMAGES

The  button on the X'Pert toolbar accesses the save options. By default, images are saved in the ScanData folder.

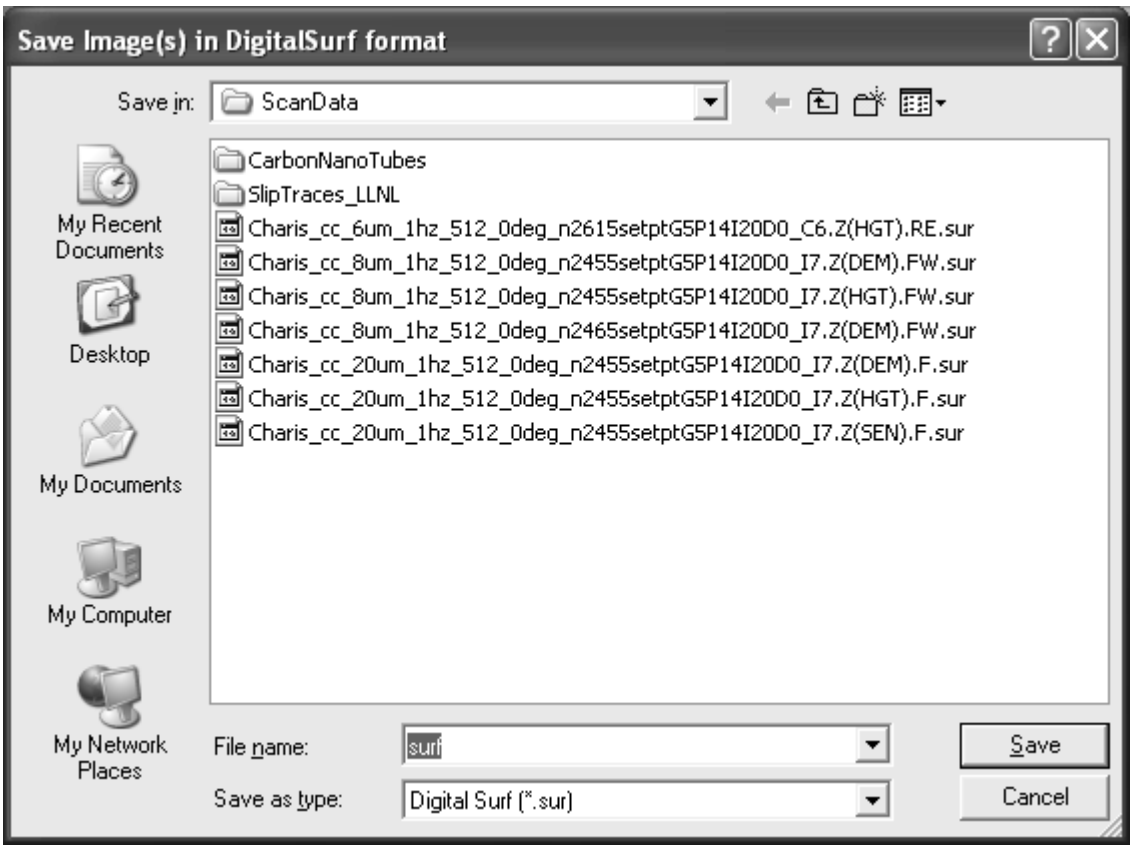
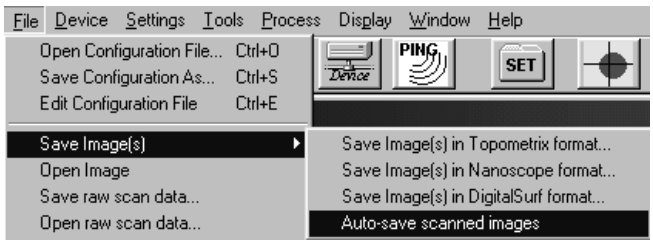
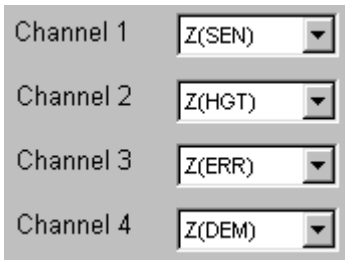


Figure 5.5. Saving images.

To save every scan that is taken, select the Auto-save scanned images option. Images will be saved in the folder that was selected the last time an image was saved.



By default, four channels are selected (in the Scan image window). So when an image is saved, a total of eight files are saved, both the forward and reverse scan data for each channel selected.



If some channels are not necessary for your application, you may want to reduce the number of active acquisition channels in order to reduce the number of files generated. To make sure the channels you are interested in are active, select Settings→Input Selects to ADC, and make sure these are listed as the primary channels. For example, if the Acq. Channels setting (in the Scan image window) is set to "2," the signals designated as Channel 1 and Channel 2 will be used.

Appendix:
A Guide to AFM Image Artifacts

INTRODUCTION

All measurement instrumentation used by scientists and engineers for research development and quality control generates results that may have artifacts. This appendix serves as a guide to identify common artifacts that occur in AFM images. It is organized into the following sections, covering the four primary sources of AFM artifacts:

- Probes
- Scanners
- Image Processing
- Vibrations

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 of interest on the sample are in the 100 nanometer range, a probe with a diameter as large as 10 nanometers 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.

Following are some of the more common probe artifacts.

SURFACE FEATURES APPEAR TOO LARGE

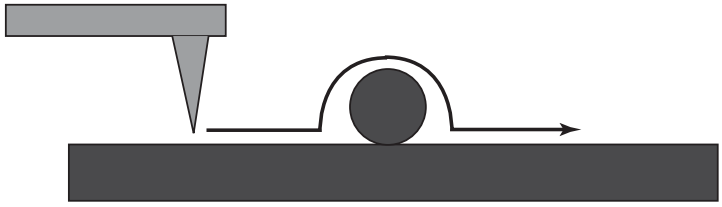


Figure A.1. AFM probe scanning over a spherical surface feature.

Often the size of surface features, such as nanotubes and nanospheres, look larger than expected. In the measurement illustrated in Figure A.1, the side of the probe will cause a broadening of features in the image. However, the height of the feature is correct when measured by a line profile.

In Figure A.2, the line profile of the image shows a diameter of 92 nm and a height of 8 nm. The broadening in the image is caused by the shape of the probe.

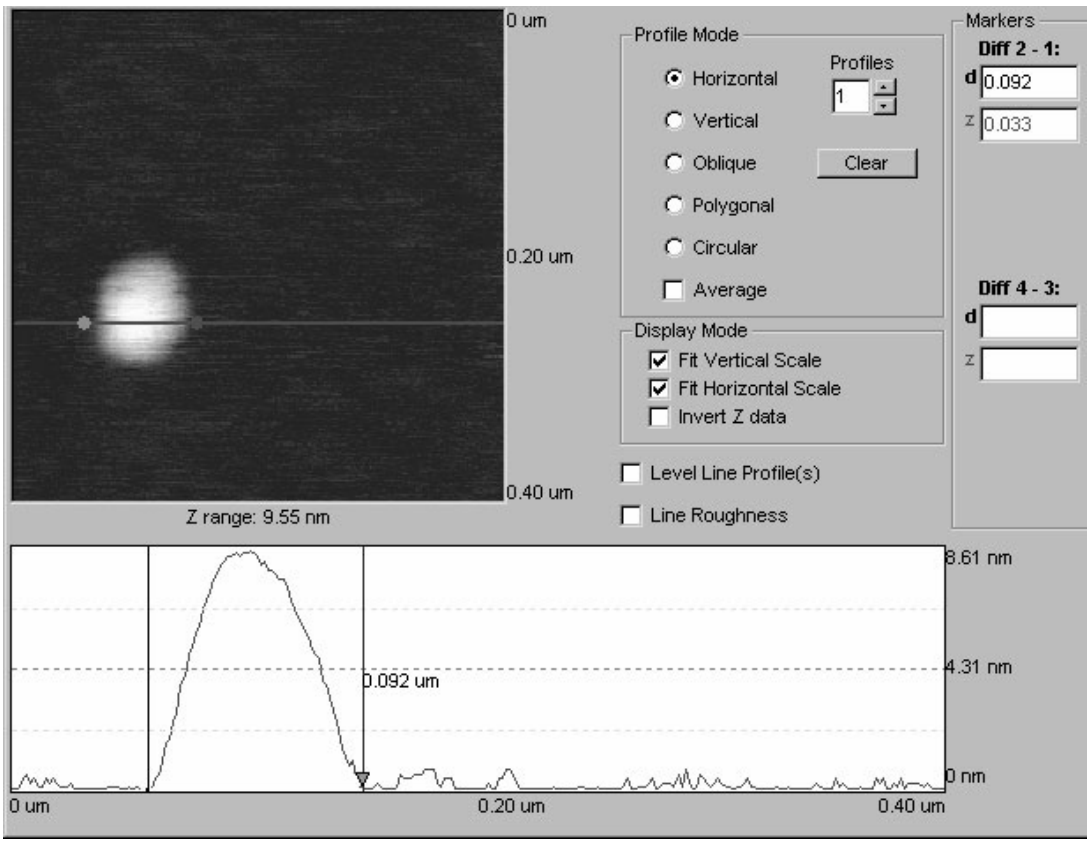


Figure A.2. AFM image and line profile of an 8 nm diameter sphere. Scan size: 400 X 400 nm.

SUB-SURFACE FEATURES APPEAR TOO SMALL

When the probe measures a feature below the sample surface, the size of the feature can appear too small. The line profile in these cases is established by the geometry of the probe rather than the geometry of the sample. For example, in the measurement illustrated in Figure A.3, the width of the probe prevents it from reaching the bottom of the feature.

Figure A.3. AFM probe scanning over a depression in the surface topography.

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 do not reach the bottom of the features.

In Figure A.4, the SEM image shows the sides of the squares in the test pattern to be equal. In the AFM image, because the probe is not sharp, the squares appear much smaller than they are, and as rectangles, not squares.

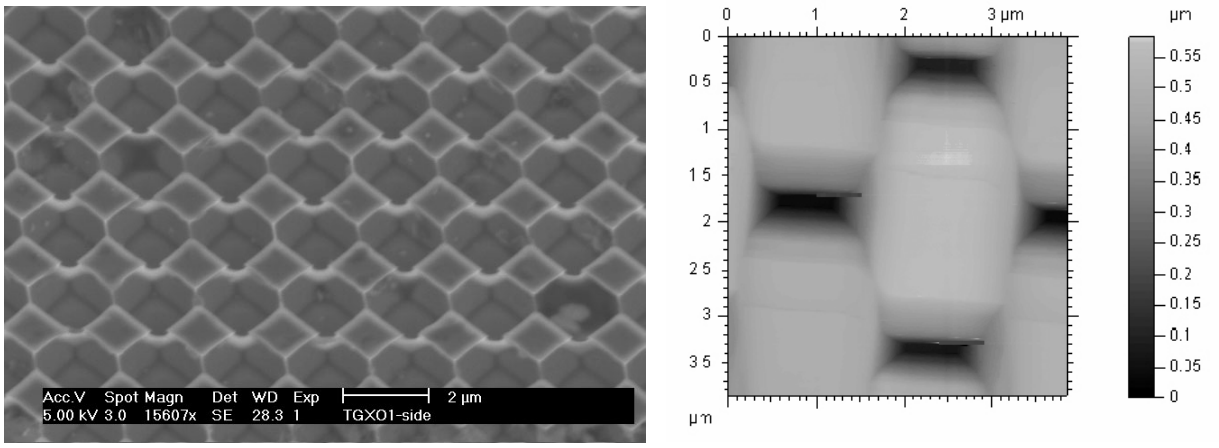


Figure A.4. SEM (left) and AFM (right) images of a test pattern of squares (NT-MDT TXO1).

STRANGELY SHAPED OBJECTS

If the probe is broken or chipped, the resulting image may have strangely shaped objects that are difficult to explain. The chipped probe in Figure A.5 follows the surface geometry in a way which creates an image with a substantial artifact.

Figure A.5. Chipped AFM probe scanning over a sample surface.

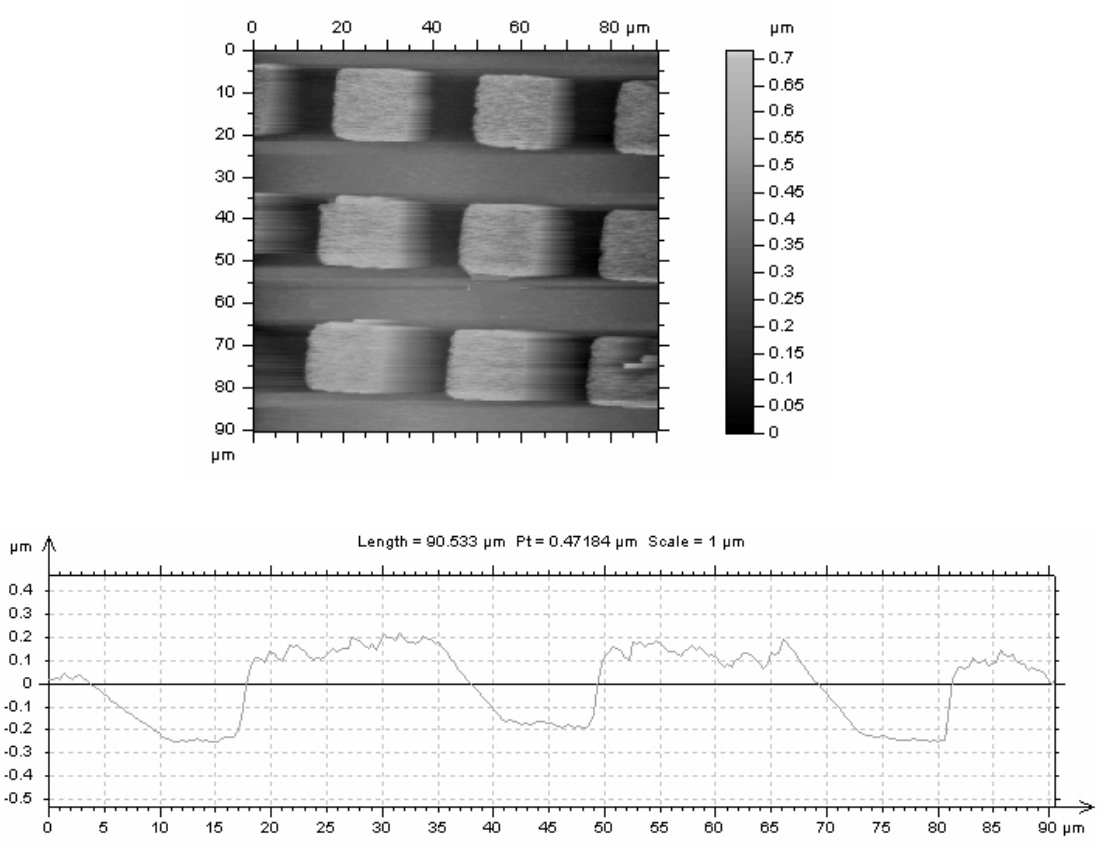


Figure A.6. AFM image of a semiconductor test pattern and the line profile showing an artifact. Scan size: 91 X 91µm.

The dark right edges in the image in Figure A.6 would indicate that the tip was scanned at a large angle to the surface, as described below (page 96). However, the probe-sample angle would have to be extreme to explain this artifact. The artifact can be easily seen in the line profile.

REPEATING STRANGE PATTERNS

If the surface features are much smaller than the probe, it is possible to see large numbers of repeating patterns in the image. The patterns will often appear as triangles, especially if silicon probes are used.

Figure A.7 shows AFM images of colloidal gold particles that reflect the shape of the tip rather than their own geometry. Compare the AFM images of the nanospheres, which should be perfect spheres, with the SEM images of the tips used to take the AFM images. Because the chipped tips are much larger than the nanospheres, the geometry of the probes is reflected in the AFM images.

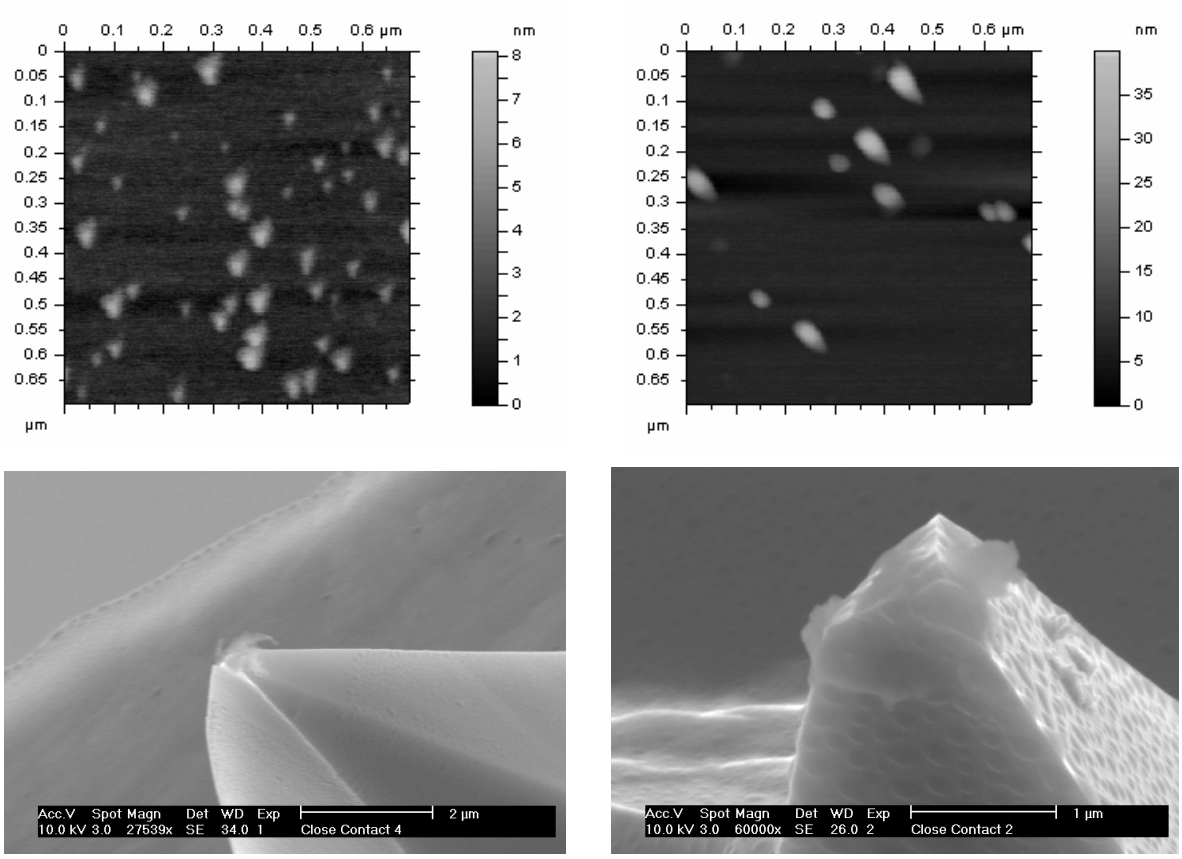


Figure A.7. AFM images of nanospheres (top) and SEM images of the probes used (bottom). Diameter of nanospheres: 5 nm (left) and 28 nm (right). Scan size: 700 nm X 700 nm.

SCANNER ARTIFACTS

The scanners in an atomic force microscope that move the probe 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. Furthermore, the piezoelectric ceramics exhibit hysteresis effects caused by self-heating. Artifacts can also be introduced into images due to the geometry of the scanner and the positioning of the scanner relative to the sample.

PROBE-SAMPLE ANGLE

If the surface features are much smaller 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 tip should be perpendicular to the surface.

Figure A.8. A sharp probe scanning at an angle.

In the measurement illustrated in Figure A.8, the probe is much sharper than the feature, so the image should be correct. 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 it is perpendicular. In some AFM microscopes, the probe is designed to be at a 12 degree angle with respect to the sample. Some microscopes do not have mechanical adjustments to control the probe-sample angle.

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. Also, the motion of the scanners must 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.

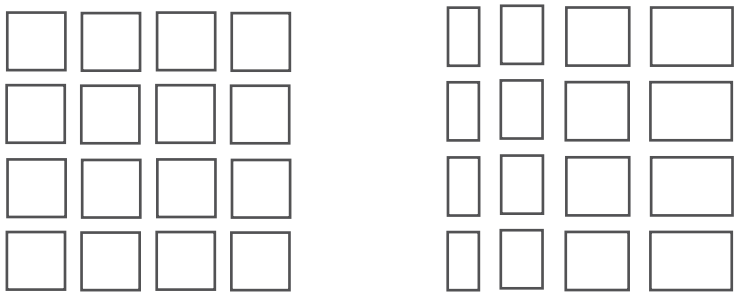


Figure A.9. A test pattern of squares (left) will appear severely distorted (right) if the piezoelectric scanner in the AFM is not linear.

The AFM image of the test pattern in Figure A.10is very linear. It appears as it should, with consistent spacing of the squares on all sides.

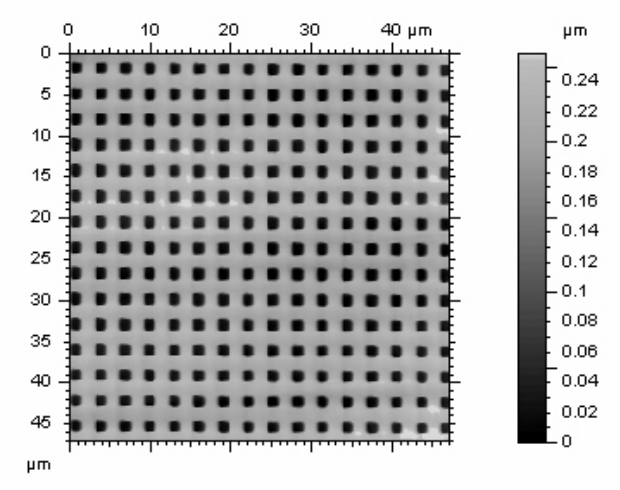


Figure A.10. Linear AFM image of a test pattern.

Once the scanner is properly linearized, it is also critical that it be calibrated. If it is linear but not calibrated correctly, the X-Y values measured from line profiles will be incorrect.

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.

Z CALIBRATION/LINEARITY

Accurate AFM height measurements depend on the piezoelectric ceramics in the Z axis being both linear and calibrated. If the microscope is calibrated at only one height, the height measurements will only be correct if the relationship between the measured Z height and the actual Z height is linear.

Figure A.11. Z calibration at only one point.

The graph in Figure A.11 shows the relationship between an actual Z height and a measured Z height in an AFM. In cases where only one calibration point is measured, as represented by the grey circle, the Z ceramic is assumed to be linear, as shown by the straight line. However, as is often the case, the ceramic is nonlinear, as shown by the bowed line. When this is the case, the microscope will measure incorrect Z heights unless the feature being measured is close to the calibration measurement.

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.

In cases where a background bow and background tilt are larger than the features of interest, the background must be subtracted from the image. This is often called “leveling” or “flattening” the image. Typically, leveling the image should make the desired features clearly visible.

The piezoelectric scanner is often supported at the top by a mechanical assembly, as shown in Figure A.12, and the motion of the probe is therefore 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.

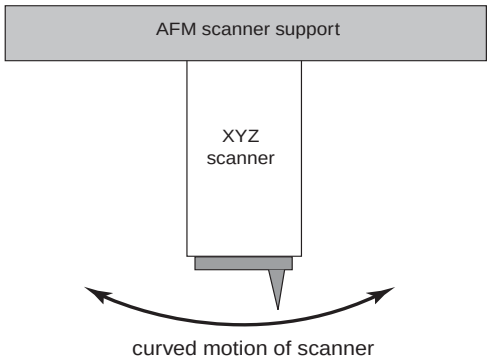


Figure A.12. Nonlinear Z scanner motion.

In Figure A.13, the bow introduced into the image is seen at the edges. The line profile across the image shows the magnitude of the bow.

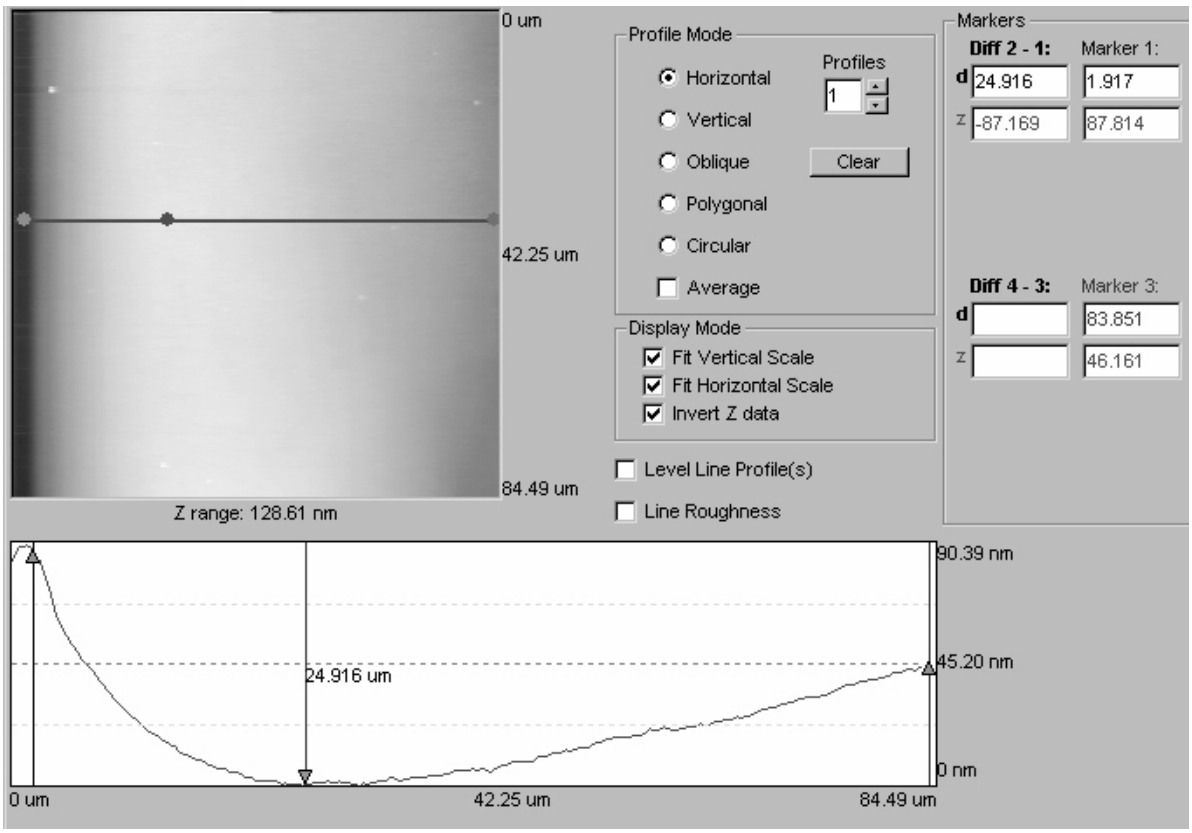


Figure A.13. Bow in AFM image and line profile of a flat piece of silicon. Scan size: 85 X 85 µm.

Z EDGE OVERSHOOT

Hysteresis in the piezoelectric ceramic that moves the probe in the Z direction can cause what is known as “edge overshoot.” This problem is most often observed when imaging micro-fabricated structures such as patterned Si wafers or compact discs. The effect can visually improve the images by making the edges appear sharper. However, a line profile of the structure shows errors.

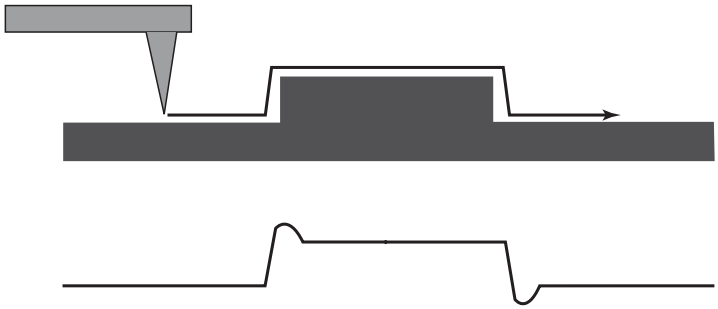


Figure A.14. Overshoot in scan (top) is apparent in the line profile (bottom).

Any overshoot that occurs as the probe is scanned over a surface feature would be apparent in the line profile of the resulting image, at the leading and trailing edges of the structure, as shown in Figure A.14 and Figure A.15.

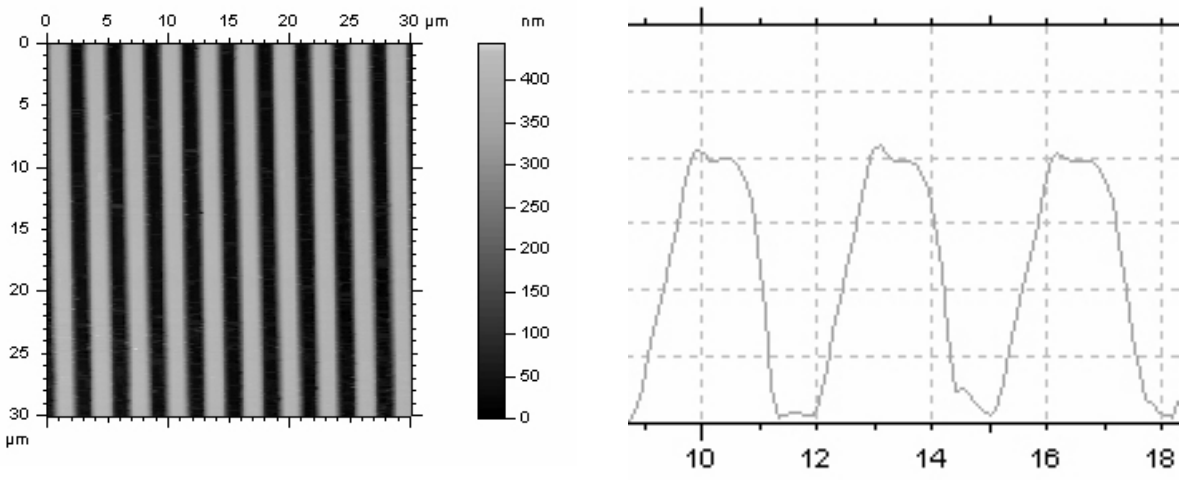


Figure A.15. This AFM image of a test pattern appears to have no artifacts, but the line profile shows overshoot at the top of each line.

SCANNER DRIFT

Drift in AFM images can be due to thermal drift in the piezoelectric scanner and the susceptibility of AFMs to external temperature changes. In AFM imaging, it is common to zoom in to a small area of a scanned region and take a new scan in order to get a higher magnification. The most common type of drift shows up as distortion at the beginning of such a scan, as shown in Figure A.16. Drift artifacts are most easily observed when imaging test patterns: lines that should appear straight have curvature.

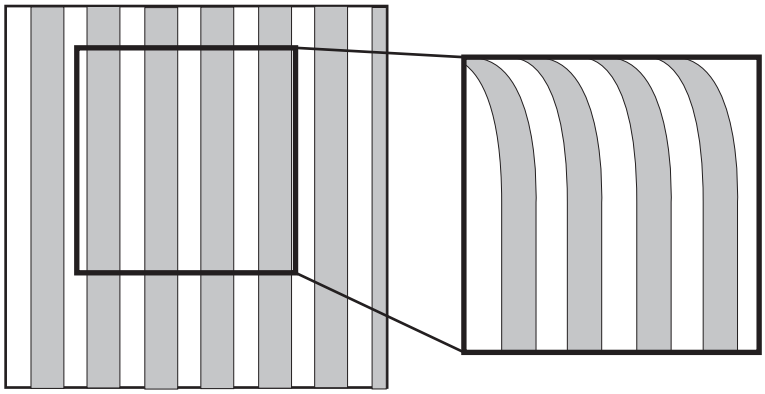


Figure A.16. Distortion due to drift in the initial part of a scan of a zoomed-in area.

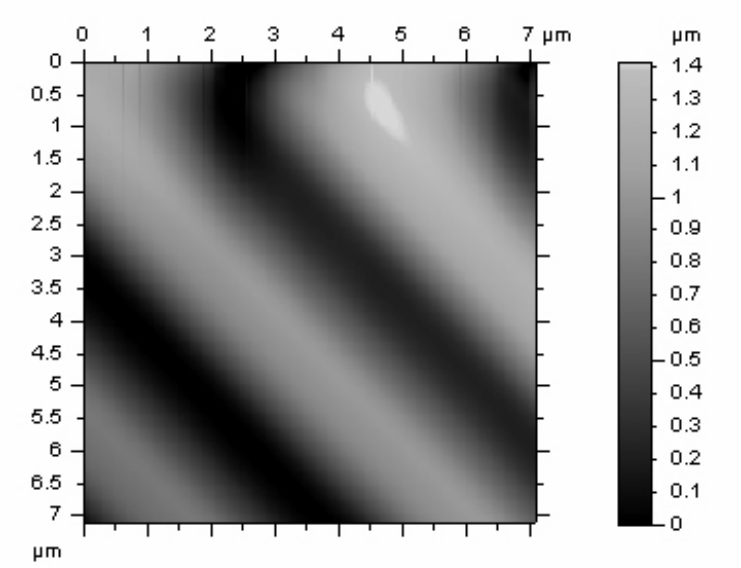


Figure A.17. Zoomed image showing a distortion at the beginning of the scan (scan angle: 45°).

X-Y Angle Measurements

Errors in the horizontal measurements in an image can result if the motion generated by the X-Y scanner is not orthogonal. 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 image. The lines drawn on the test pattern image in Figure A.18 show no measurable cross-talk between the X and the Y axis.

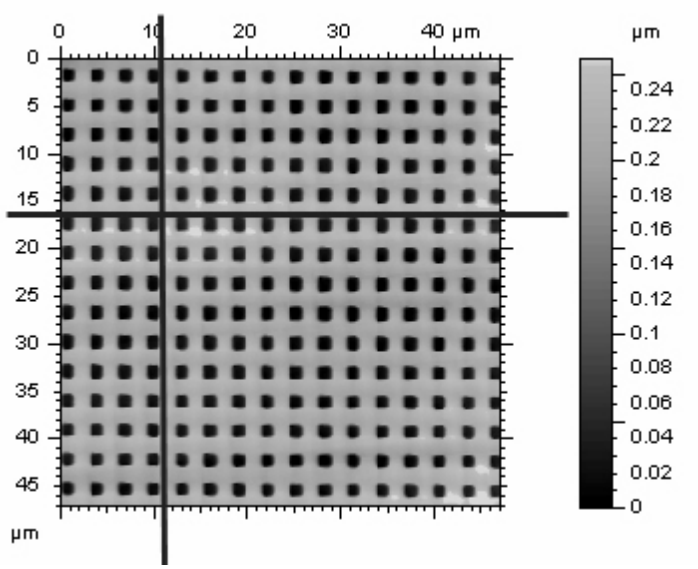


Figure A.18. AFM image of a test pattern with lines drawn to measure any error in orthogonality.

Z Angle Measurements

Mechanical coupling between the piezoelectric ceramics that move the probe in the Z direction and those that move the probe in the X or Y directions can cause substantial errors when trying to measure side wall angles. This error can best be measured with a sample that has repeating triangular structures, as illustrated in Figure A.19 and Figure A.20.

Figure A.19. Asymmetry caused by mechanical coupling between Z and X or Y piezoelectric ceramics.

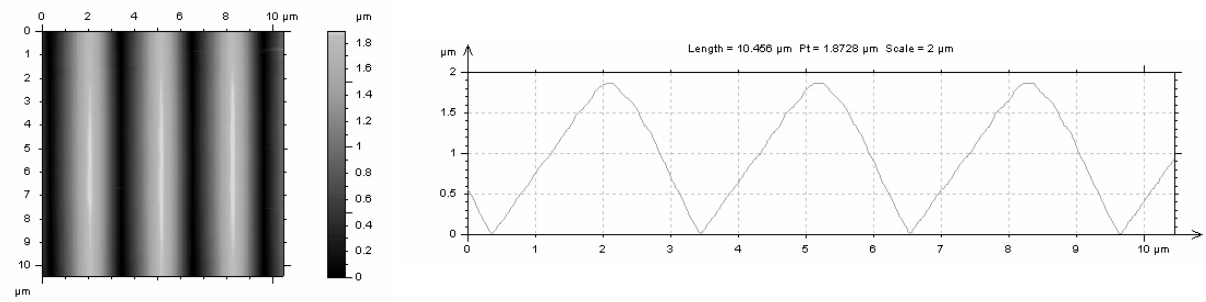


Figure A.20. AFM image of a sample with a repeating triangle pattern and the extracted line profile.

IMAGE PROCESSING

This section presents some of the common artifacts that can be introduced into AFM images by image processing software. Almost all AFM images require some image processing before viewing or analysis, and 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.

LEVELING

Most AFM images have some tilt and bow caused by the scanner or stage configuration (as described above on page 98). A number of background subtraction options are possible. The two most common types are:

- Line-by-line leveling: 0 to 4^{th} order
- Plane Leveling: 0 to 4^{th} order

Image processing 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.

A typical leveling routine is illustrated in Figure A.21. In the original image (A), before any image processing, tilt is easily recognized: the right side of the image appears darker than the left side. The second image (B) is the result of line-by-line leveling with a first-order background correction. The dark band is caused by the image processing and is not a real structure. The third image (C) was derived by excluding particles from the background subtraction process.

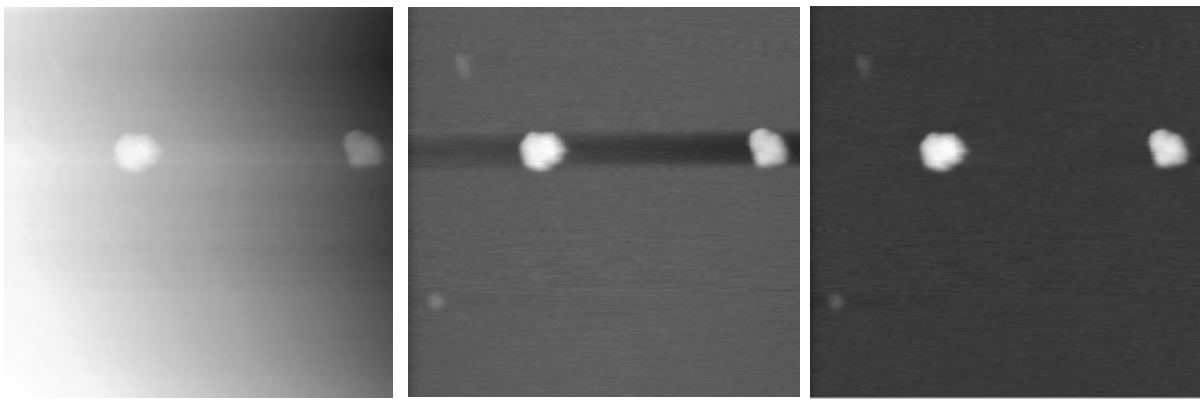


Figure A.21. Leveling of a 1.6 X 1.6 µm AFM image of nanospheres.

HIGH-PASS FILTER

A high-pass filter is often used to "smooth" data before it displays. In images with substantial high-pass filtering, the dimensions like the step in Figure A.22 can appear distorted. The amount of distortion depends on the amount of filtering applied to the image. Other image artifacts can appear as a sharpness at the edge of steps.

Figure A.22. Image distortion due to high-pass filtering.

FOURIER FILTERING

Fourier filtering can easily introduce periodic structures into images. For example, an image of "white noise" can be filtered to give periodic structure that looks like atomic structure.

MATRIX-FILTER SMOOTHING

Matrix filtering is a very effective way of "smoothing" images and removing noise. However, the filtering process often reduces the resolution. As a rule of thumb, if the image has no noise in it, the data has probably been compromised. The example in Figure A.23 shows how filtering can reduce the noise, as shown in the line profiles, but also cause the shape of the nanospheres to be altered.

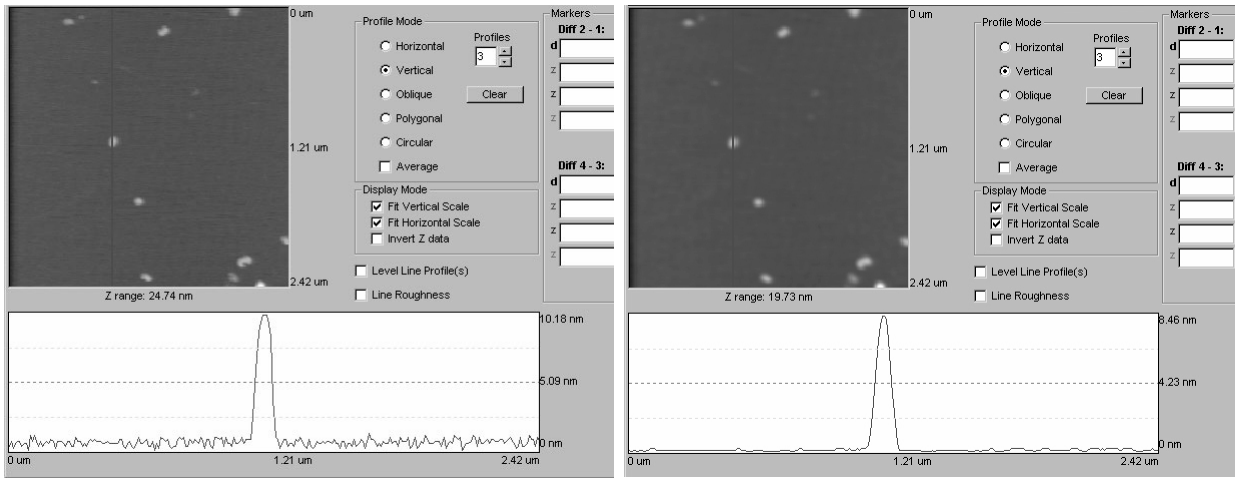


Figure A.23. AFM image of nanospheres before and after matrix smoothing.

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.

The image in Figure A.24 was derived from an image with substantial noise. Filtering has added the "nodules," which make it seem like a much higher resolution image.

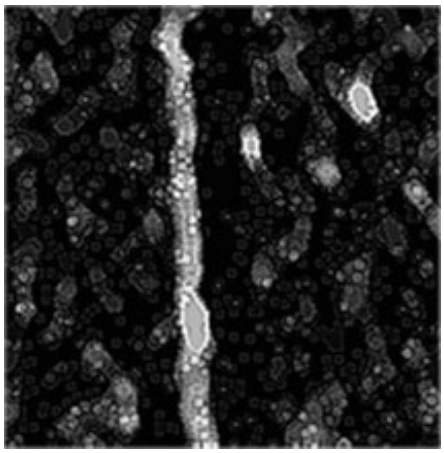


Figure A.24. AFM image of a nanotube showing "nodules" due to filtering. Scan size: 850 X 850 nm.

VIBRATIONS

Vibrations in an AFM's operating environment can cause the probe to vibrate, resulting in image artifacts. Typically, the artifacts appear as oscillations. Both floor and acoustic vibrations can excite vibrational modes in an AFM and cause artifacts.

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.

ACOUSTIC VIBRATIONS

Sound waves can cause artifacts in AFM images. The source of the sound may be from an airplane going over the building or from the tones in a person's voice. The images and line profiles in Figure A.25 illustrate the effects of noise derived from a person talking in the same room as the microscope.

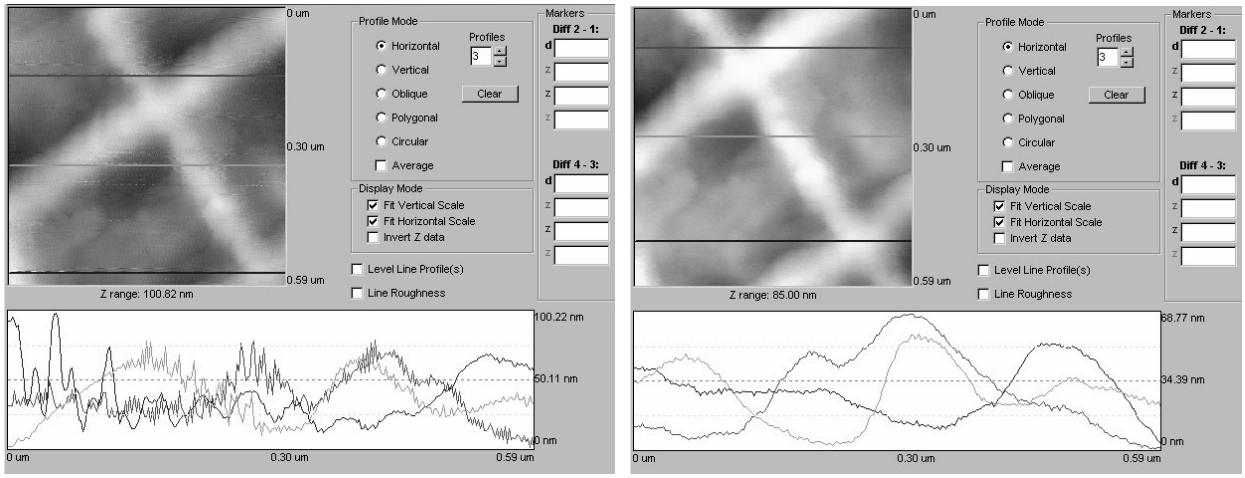


Figure A.25. High resolution images of a test grid with acoustic noise present in the room (left) and without noise (right).

OTHER SOURCES

ELECTRONICS

Faulty electronics can be a cause of artifacts in AFM images. Most often, these appear as oscillations or unexplainable repeating patterns. Electronic ground loops and broken components are usually the source of electronic noise. The electronic noise in the image in Figure A.26 was the result of not having a ground wire attached to the stage. The artifact is identified by the oscillations.

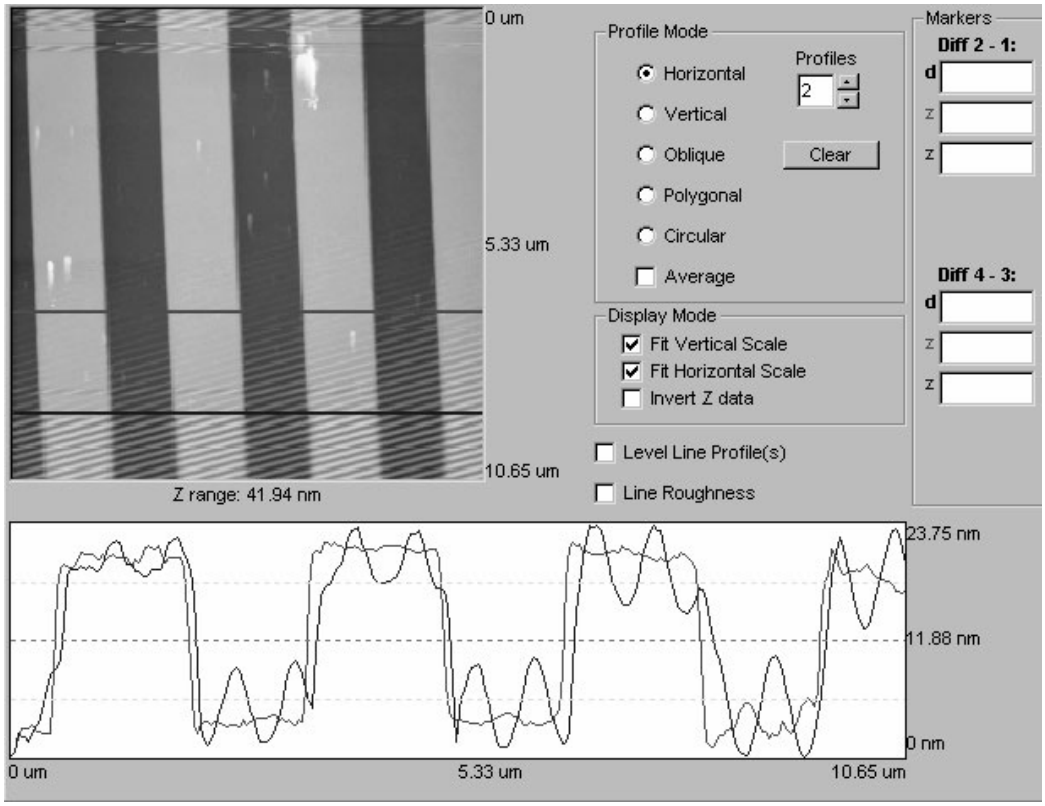


Figure A.26. Test pattern image with electronic noise at the top and bottom of the scan.

SURFACE CONTAMINATION

Substantial contamination at the sample surface, such as a fingerprint or oil film, can cause AFM image artifacts. Such artifacts appear as streaks on the image, as seen in the top of the image in Figure A.27. Streaks tends to appear in areas of the sample surface having "sharp" features and edges. Often the streaking can be reduced or even eliminated by cleaning the sample with a high purity solvent.

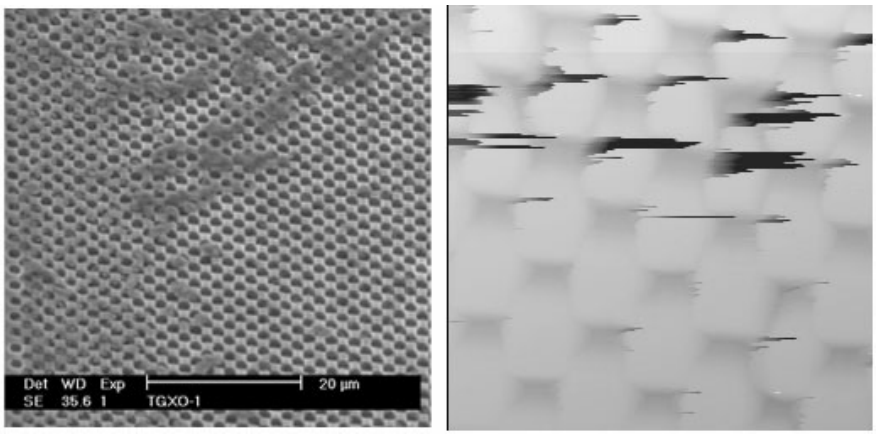


Figure A.27. SEM image (left) and AFM image (right) of a contaminated test pattern.

VACUUM LEAKS

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