

Keysight 5500 Scanning Probe Microscope

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Safety Considerations

- **General** - This product and related documentation must be reviewed for familiarization with these safety markings and instructions before operation.

This product is a safety Class I instrument (provided with a protective earth terminal).

- **Before Applying Power** - Verify that the product is set to match the available line voltage and the correct fuse is installed. Refer to instructions in "**Facility Requirements**" on page 61 of the manual.
- **Before Cleaning** - Disconnect the product from operating power before cleaning.
- **Safety Earth Ground** - An uninterrupted safety earth ground must be provided from the main power source to the product input wiring terminals or supplied power cable.

Specifications

Environmental Conditions

Temperature (Operating): 5 to 40 °C

Temperature (Non-operating): -40 to 70 °C

Relative Humidity (Operating): 15 to 95 % non-condensing

Altitude: 2000 m

Power Requirements

100/120/220/240 VAC, 50/60 Hz

Mains supply voltage fluctuations are not to exceed 10 % of the nominal supply voltage.

NOTE

These specifications apply to the Keysight 5500 system, and do not guarantee the function of an experiment (including the cantilever) in these conditions.

Equipment Class I, Pollution Degree 2, Installation Category II.

This equipment is for indoor use only.

When the product is subjected to 8 kV air discharge or 4 kV contact discharge in accordance with IEC 61000-4-2, interruption of the laser output may occur.

If this happens, laser power must be re-cycled in order to resume normal operation.

CAUTION

Stop using the scanner if the scanner cable insulation is damaged in order to avoid electrical shock. Have it repaired or replaced by the factory.

Voltage and Laser Safety Information

The 5500 SPM will be marked with the following Instruction Manual symbol when it is necessary for the user to refer to this User's Guide:



The following symbol indicates hazardous voltages:



This system is designed to be used with a Class II or Class III diode laser with an output of up to 1 mW of visible radiation at 670 nm or 980 nm. The aperture in the AFM scanning head is labeled with the logotype (shown below). DO NOT stare directly into the laser beam. To ensure safe operation, the scanner must be operated and maintained in accordance with the instructions included with the laser. The laser must only be powered by a controller that includes an on/off switch, such as the Keysight SPM Controller. DO NOT attempt to make any adjustments to the laser, the laser's electronics, or optics. If laser malfunction is suspected, immediately return the scanner to Keysight Technologies for repair or replacement; there are no user-serviceable parts.

WARNING

Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous light exposure. Furthermore, the use of optical instruments with this product may increase eye hazard.

In accordance with federal FDA requirements, one of the following laser precautions is affixed to the scanner:



Power Supply

It is not necessary to open the Keysight AFM Controller to make changes to the power supply. However, the power cord should always be unplugged before making any adjustments to the power source. The Keysight SPM Controller has several different power supply options.

Procedure for Changing Input Voltage

- 1 Unplug the power cord from the Keysight AFM Controller.
- 2 Remove the fuse holder located on the back of the controller.
- 3 Underneath where the fuse holder was located is the input voltage control switch. Pull out the switch and rotate it to the desired input voltage 100/120/220/240 V.
- 4 Reinsert the voltage switch with the desired voltage.
- 5 Replace the fuse holder.

Piezo Scanner Precautions

Piezo scanners are, by nature, very FRAGILE pieces of equipment. The piezo material that does the scanning is a ceramic and is consequently quite easily broken. Dropping a piezo scanner will result in damage to the scanner that can only be repaired by completely replacing the scanner piezo core. This can be an expensive and time-consuming process and so it is advised that the utmost care is used when handling the scanners. Keysight Technologies recommends that the scanners be stored in the padded scanner case that was supplied with the scanner and that the scanner be kept in a dry environment when not in use. Piezo scanners also perform better with consistent use. If a scanner is not used for some time it may require a short period of use before the scan range is stable and the calibration is correct. It may also be necessary to re-calibrate the scanner from time to time. The calibration can be

verified using a calibration standard, and adjustments can be made using the calibration tools.

General Care Requirements

SPM equipment is sensitive scientific equipment. Care must be used when handling all parts. When removing scanners from the microscope ensure that all cable connections to the scanner are disconnected. This includes cables for photo-diode detectors. Also, the photo-diode detector should be removed from the scanner prior to the removal of the scanner from the microscope. All equipment, especially the sample plates and scanner nose modules should be kept clean and free from contamination when not in use. It is recommended, to prolong the life of these items, that after use all sample plates and noses are cleaned thoroughly and dried off prior to storage. Cleaning can be done using an organic solvent. Please refer to the appropriate sections of the manual for further information regarding the proper cleaning of equipment.

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Declaration of Conformity



Agilent Technologies

DECLARATION OF CONFORMITY
According to ISO/IEC Guide 22 and CE N/CENELEC EN 45014



Manufacturer's Name: Agilent Technologies, Incorporated
Manufacturer's Address: 5301 Stevens Creek Boulevard
Supplier's Address: Santa Clara, CA 95051
USA

Declares under sole responsibility that the product as originally delivered

Product Name: PicoPlus – Atomic Force Microscope
Model Number: Series 5500
Product Options: This declaration covers all options of the above products

complies with the essential requirements of the following applicable European Directives, and carries the CE marking accordingly:

- The Low Voltage Directive 73/23/EEC, amended by 93/68/EEC
- The EMC Directive 89/336/EEC, amended by 93/68/EEC

and conforms with the following product standards:

EMC	Standard	Limit
	IEC 61326-1:1997+A1:1998 / EN 61326-1:1997+A1:1998	
	CISPR 11:1990 / EN 55011:1991	Group 1 Class A
	IEC 61000-4-2: 1995+A1: 1998 / EN 61000-4-2:1995	4 kV CD, 8kV AD
	IEC 61000-4-3: 1995 / EN 61000-4-3: 1995	3 V/m, 80-1000MHz
	IEC 61000-4-4: 1995 / EN 61000-4-4: 1995	0.5 kV signal lines, 1 kV power lines
	IEC 61000-4-5: 1995 / EN 61000-4-5: 1995	0.5 kV line-line, 1kV line-ground
	IEC 61000-4-6: 1995 / EN 61000-4-6: 1995	3 V, 0.15-80 MHz 1 cycle, 100%
	IEC 61000-4-11: 1994 / EN 61000-4-11: 1994	Dips: 30% 10ms; 60% 100ms
		Interrupt: > <u>95%@5000ms</u>

Canada: ICES-001:1998
Australia/New Zealand: AS/NZS 2064.1

This product was tested in a typical configuration with Agilent Technologies test systems

Safety IEC 61010-1:2001 / EN 61010-1:2001
IEC 60825-1:1993+A1:1997+A2:2001
EN 60825-1:1994, Class 2 Laser Product
USA: 21CFR 1040.10+1040.11, Class II
Canada: CSA C22.2 No. 1010.1:1992

Supplementary Information:

This DoC applies to above-listed products placed on the EU market after:

25 August 2006

Date

Randall White

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Product Regulations Manager

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The Keysight 5500 SPM is the ideal multiple-user research system for Scanning Probe Microscopy (SPM). As the high-performance Atomic Force Microscope (AFM) flagship of Keysight's product line, the 5500 SPM provides a wealth of unique technological features, including precision temperature control and industry-leading environmental control.

The Keysight 5500 SPM offers features and software for research in materials science, polymers, nanolithography and general surface characterization. With excellent ease of use, the 5500 SPM also affords educators an unprecedented opportunity to introduce students to AFM technology.

Overview of Keysight SPM System

The main component of the Keysight 5500 SPM system is the microscope (Figure 1), which includes the X/Y motion controls, scanner, high-resolution probe/tip, and detector. The control system for the microscope includes, at minimum, a high-speed computer, AFM controller and Head Electronics Box. Optional components include additional electronics, specialized scanners and probes for particular SPM techniques, and an environmental enclosure to control acoustic and vibration noise.



Figure 1 The Keysight 5500 SPM microscope, shown with optional environmental chamber

In this User's Guide we will begin with a brief introduction to Scanning Probe Microscopy techniques. The sections that follow will show you how to handle the 5500 SPM components and how to image in the available modes.

SPM Basics

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

An SPM is used to investigate sample properties at or near the sample surface; that is, immediately beneath the surface (typically several nanometers deep) and immediately above the surface (typically several tens of nanometers high).

In SPM techniques, the sharp probe (tip) is scanned across a sample surface, or the surface is scanned beneath the tip (Figure 2). Interactions between the tip and sample are detected and mapped. Different techniques sense different interactions, which can be used to describe surface topography, adhesion, elasticity, electrostatic charge, etc.

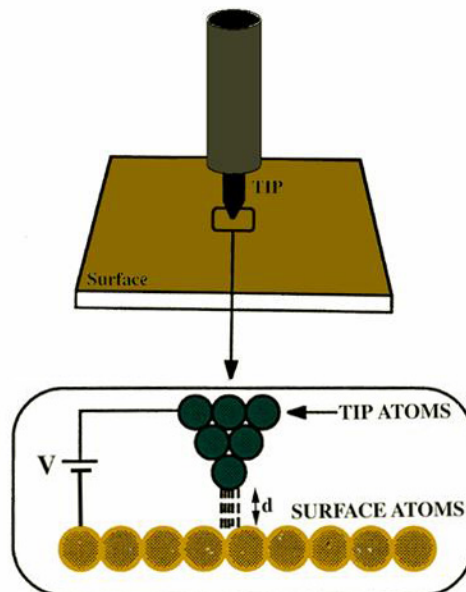


Figure 2 Scanning Probe Microscopy diagram

The small size of the probe tip is key to the SPM's high resolution. However, its small size also means that the tip must be scanned in order to image a significant area of the sample. SPM techniques use "raster scanning," in which high resolution actuators, usually made of piezoelectric materials, move the probe across the sample and back over each line of the image area. For each X/Y coordinate pair, the interaction of the tip and sample is recorded as one data point. The

collection of data points is then synthesized into the “SPM image,” a 3-dimensional map of the surface characteristic being examined.

The most common SPM images are topography images, in which the third dimension, Z, for any given X/Y coordinates, is the relative height of the sample surface. This interpretation implies that the sharp probe does not deform the sample surface—the harder the sample surface, the more accurate is this interpretation. In other words, the tip follows the height variations of hard surface with higher fidelity than it does soft surfaces.

Topography measurements are in general calibrated against height standards. Therefore, topography images may be compared for quantitative information, provided the systems have been correctly calibrated and operated, and that the data is properly interpreted.

In other types of SPM images, the third dimension is a measure of the relative strength of a detectable interaction between the probe and sample. The image is usually recorded simultaneously with, and displayed along side, the topography image of the same sample area. This helps reveal any correlation between topography and the interaction.

In some instances, the signal from the SPM’s detector is mapped directly; for example, the deflection of the probe cantilever, or the current through a metal tip. In other instances, the signal from the detector serves as the input of a feedback system which attempts to maintain the detector signal at a user-defined setpoint. The output of the feedback system can then be mapped to construct the image.

SPM can also be used for “non-imaging techniques,” or “nano-manipulation,” in which the probe is used to modify the sample surface. For example, one can use the probe or tip to rearrange nanometer-scale objects physisorbed on that surface. Essentially, the tip serves as a nano-scale finger to interact with the sample.

Nano-manipulation is sometimes performed in the plane of the sample surface (in-plane) and sometimes at right angles to this plane (out-of-plane nano-manipulation). An example of out-of-plane nano-manipulation is attaching the probe tip to the end of a macromolecule on the sample surface, and pulling the molecule so that its secondary or tertiary structure unfolds. This is now an extremely active area of research, with applications extending to fields as diverse as drug discovery and composite materials design.

SPM Techniques

Scanning Tunneling Microscopy (STM)

The earliest, widely-adopted SPM technique was Scanning Tunneling Microscopy (STM). In STM, a bias voltage is applied between a sharp, conducting tip and the sample. When the tip approaches the sample, electrons “tunnel” through the narrow gap, either from the sample to the tip or vice versa, depending on the bias voltage. Changes of only 0.1 nm in the separation distance cause an order of magnitude difference in the tunneling current, giving STM remarkably high precision. The basic STM schematic is shown in [Figure 3](#).

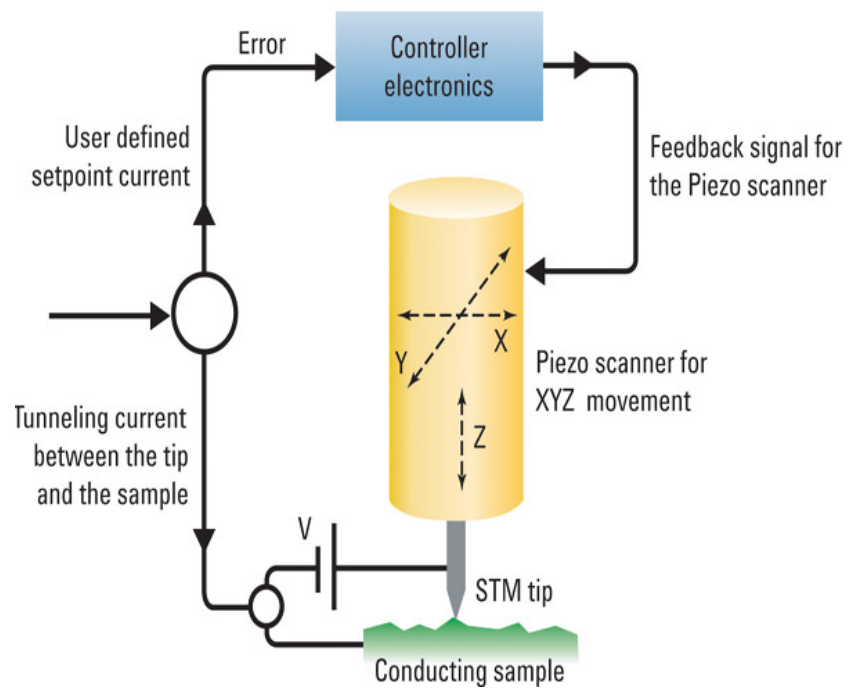


Figure 3 Basic STM schematic

STM can image a sample surface in either *constant current* or *constant height* mode, as described in [Figure 4](#). In *constant height* mode, the tip remains in a constant plane above the sample, and the tunneling current varies depending on topography and local surface properties. The tunneling current measured at each location constitutes the image. The sample surface, however, must be relatively smooth in order for the system to acquire useful information.

In *constant current* mode, a feedback loop is used to adjust the height of the tip in order to hold the tunneling current at a setpoint value. The scanner height measured at each location is then used to map the surface

topography. Because the feedback response requires time, constant current mode is typically slower than constant height mode. However, greater variations in height can be accommodated.

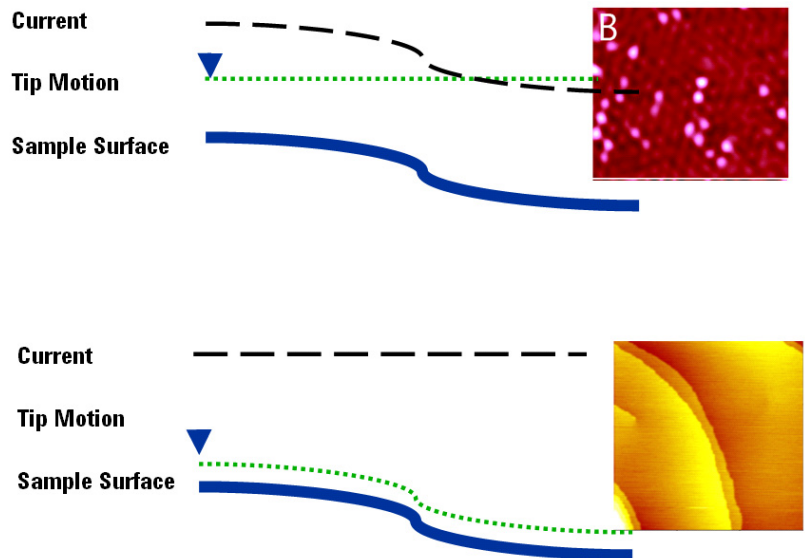


Figure 4 Constant Height mode STM (above) is faster but is limited to smooth surfaces; Constant Current mode (below) is capable of mapping larger variation in Z

For electron tunneling to occur, both the sample and tip must be conductive or semi-conductive. Therefore, STM cannot be used on insulating materials. This is one of the significant limitations of STM, which led to the development of other SPM methods described below.

Atomic Force Microscopy (AFM)

Atomic Force Microscopy (AFM) can resolve features as small as an atomic lattice, for either conductive or non-conductive samples. AFM provides high-resolution and three-dimensional information, with little sample preparation. The technique makes it possible to image *in-situ*, in fluid, under controlled temperature and in other controlled environments. The potential of AFM extends to applications in life science, materials science, electrochemistry, polymer science, biophysics, nanotechnology, and biotechnology.

In AFM, as shown in [Figure 5](#), a sharp tip at the free end of a cantilever (the “probe”) is brought into contact with the sample surface. The tip interacts with the surface, causing the cantilever to bend. A laser spot is reflected from the cantilever onto a position-sensitive photodiode

detector. As the cantilever bends, the position of the laser spot changes. The resulting signal from the detector is the Deflection, in volts. The difference between the Deflection value and the user-specified Set Point is called the “error signal.”

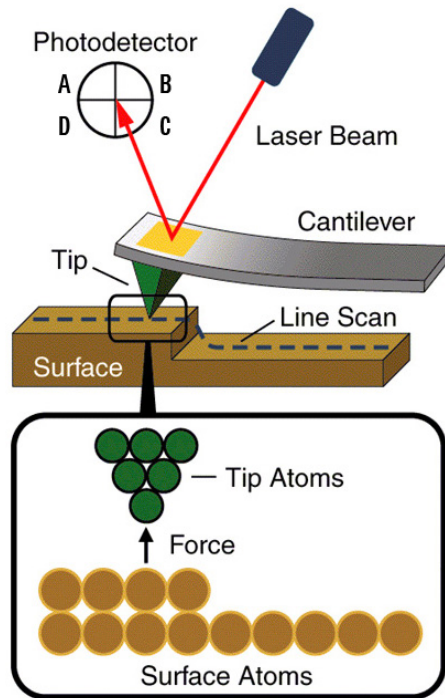


Figure 5 Basic AFM principles

Figure 6 shows the force interaction as the tip approaches the sample. At the right side of the curve the tip and sample are separated by large distance. As they approach, tip and sample atoms first weakly attract each other. This zone of interaction is known as the “non-contact” regime. Closer still, in the “intermittent contact” regime, the repulsive van der Waals force predominates. When the distance between tip and sample is just a few angstroms, the forces balance, and the net force drops to zero. When the total force becomes positive (repulsive), the atoms are in the “contact” regime. The various AFM techniques

described below, can be generally described by their function within these three domains.

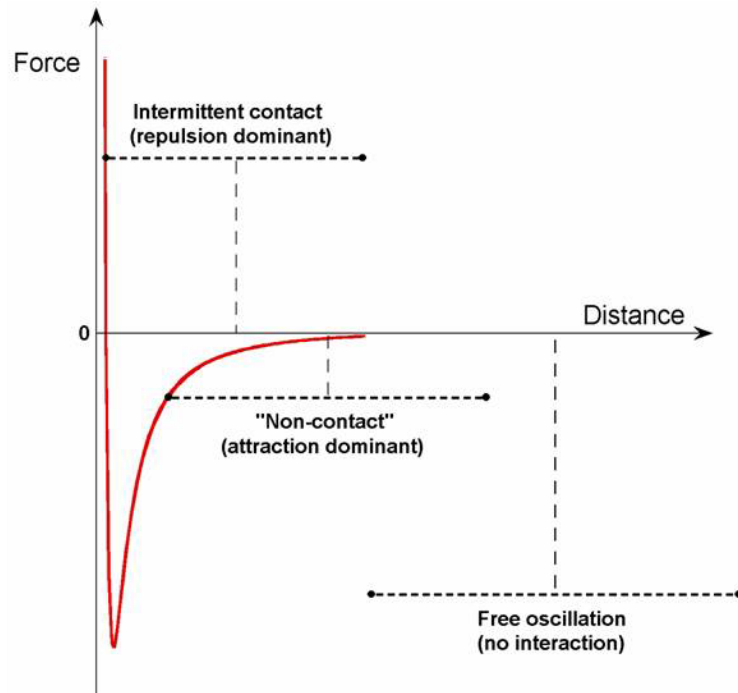


Figure 6 Zones of interaction as the tip approaches the sample

The tip-sample interaction is complicated by additional forces, including strong capillary and adhesive forces that attract the tip and sample. The capillary force arises when water, often present when imaging in the ambient environment, wicks around the tip, holding the tip in contact with the surface. As long as the tip is in contact with the sample, the capillary force should be constant because the fluid between the tip and the sample is virtually incompressible. The total force that the tip exerts on the sample is the sum of the capillary, adhesive and van der Waals forces.

The van der Waals force counters almost any force that attempts to push the atoms closer together. When the cantilever pushes the tip against the sample, the cantilever bends rather than forcing the tip closer to the sample atoms. The deflection, therefore, can be used as a reliable indicator of surface topography.

Contact Mode AFM

In Contact Mode AFM, the AFM tip is attached to the end of a cantilever with a low spring constant (typically 0.001 - 5 nN/nm). The

tip makes gentle contact with the sample, exerting from ~ 0.1 -1000 nN force on the sample.

AFM can be conducted in either *constant height* or *constant force modes*.

In *constant height* mode, the height of the scanner is fixed as it scans. For small cantilever deflections (< 500 nm) on hard surfaces, the error signal (in volts) is used to generate an image that is sensitive to small changes in topography, though actual topographic information is not obtained. *Constant height* mode is often used for generating atomic-resolution images of atomically flat surfaces, where the cantilever deflections, and thus variations in applied force, are small. It is also used for recording real-time images of changing surfaces, where high scan speed is essential.

In *constant force* mode, the error signal is used as the input to a feedback circuit which, after amplification, controls the z-height piezo actuator. The feedback circuit responds to the surface topography, keeping the cantilever deflection constant, and thus holding the total force applied to the sample constant as well. The output of the feedback circuit is used to generate the topography image.

Constant force mode is more typically used than *constant height* mode as it enables imaging of greater surface height variability. The speed of scanning is limited by the response time of the feedback circuit, however. The resolution is lower than *constant height* mode as well, due to inherent noise in the piezo feedback circuit itself.

NOTE

The signal path is actually the same for *constant height* and *constant force mode*. In both cases, the error signal from the detector is the input to the feedback loop, and the output of the feedback loop is the actual deflection signal. In *constant height* mode, however, the gain for the feedback loop is set to zero, effectively turning it off. Thus, the error signal is passed through and read directly.

Intermittent Contact AFM

Intermittent Contact Mode AFM is typically referred to as AC Mode due to the alternating contact of the tip to the surface. In AC Mode, the cantilever is driven to oscillate, typically in sinusoidal motion, at or near one of its resonance frequencies. When the cantilever and sample are close during each oscillation cycle, the tip moves through an interaction potential that includes long-range attractive and short term repulsive

components. The complex tip-sample forces cause changes in the amplitude, phase and resonance frequency of the oscillating cantilever. Thus, topography, amplitude and phase can be collected simultaneously. The phase and amplitude images may highlight physical properties that are not readily discernible in the topographic map. For example, fine morphological features are, in general, better distinguished in amplitude and phase images.

The force of the oscillating tip is directed almost entirely in the Z axis; thus, very little lateral force is developed and tip/sample degradation is minimized. This benefit also makes it possible to obtain clear images of soft samples.

A feedback system is employed to maintain the oscillation amplitude at a setpoint value. The difference between the amplitude and set point, called the “error signal,” is used as the input to the feedback system. The output of the feedback loop is amplified and drives the Z-actuator. The map of this output signal is called the “Amplitude Image,” which is typically plotted side-by-side with the topography image. The topography image is the voltage applied to the piezo required to keep the oscillation amplitude constant, multiplied by the sensitivity of the piezo in nanometers/volt.

AC Mode can operate in either the intermittent contact (net repulsive) regime or the non-contact (net attractive) regime. During intermittent contact, the tip is brought close to the sample so that it lightly contacts the surface at the bottom of its travel, causing the oscillation amplitude to drop.

The tip is usually driven by a sinusoidal force, with the drive frequency typically at or near one of the cantilever’s resonance frequencies (eigenfrequencies), and most often at the fundamental frequency. Absent any tip-sample interactions, the cantilever oscillations are also sinusoidal if the drive amplitude is small enough to keep the cantilever motion small compared with the cantilever thickness.

Two methods are used to drive the cantilever oscillation: by indirect, acoustic vibration (Acoustic Mode), or by direct vibration in a magnetic field (MAC Mode).

Acoustic AC (AAC) AFM

In Acoustic AC (AAC) Mode AFM, shown schematically in [Figure 7](#) on page 25, a piezoelectric transducer (1) shakes the cantilever holder (2) at or near its resonant frequency, typically 100 to 400 kHz. Interaction between the probe (3) and the sample reduces the oscillation

amplitude—this reduction (c.) is used as a feedback signal to maintain constant amplitude of the cantilever motion.

NOTE

Acoustic AC Mode is an option for the 5500 SPM and requires the additional Mac Mode or MAC III controller.

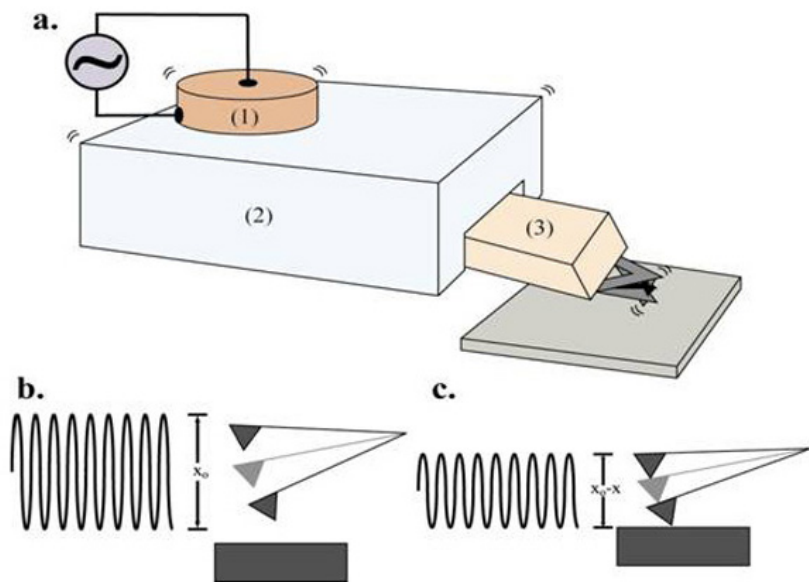


Figure 7 Acoustic AC mode (AAC) (a.) Overview (b.) The free amplitude before contact and (c.) The amount of amplitude dampening from the tip-sample interaction

Magnetic AC (MAC) Mode

In Magnetic AC (MAC) Mode AFM, the back side of the cantilever is coated with magnetic material. A solenoid applies an AC magnetic field which is used to oscillate the cantilever. (Figure 8). MAC Mode is typically cleaner and gentler than Acoustic AC Mode and is free from spurious background signals that are somewhat common when AAC Mode. The benefits are particularly pronounced when imaging in liquid.

NOTE

MAC Mode is an option for the 5500 SPM and requires the additional MAC Mode or MAC III controller.

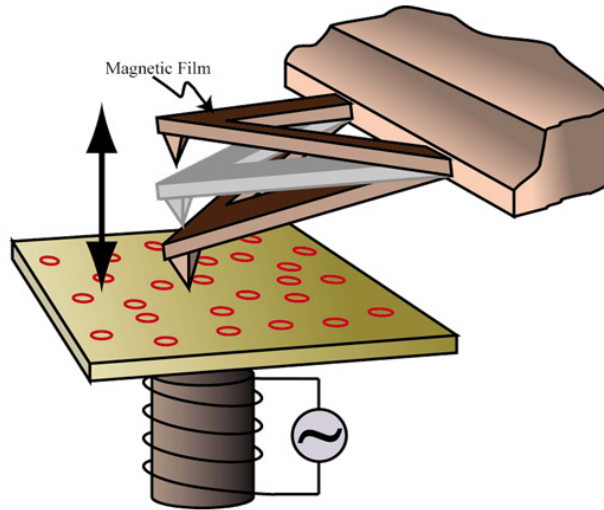


Figure 8 Magnetic AC mode (MAC mode)

Top MAC Mode

In standard MAC Mode the magnetic coil is located in the sample plate, below the sample. A variant of MAC Mode, known as Top MAC, places the drive coil above the cantilever. This enables MAC Mode to be used with or without a sample plate, for large samples, or for samples which tend to dissipate the magnetic field enough to affect the resolution of regular MAC Mode.

NOTE

Top MAC Mode is an option for the 5500 SPM and requires the additional MAC Mode or MAC III controller, as well as a Top MAC nose assembly.

Current Sensing Mode (CSAFM)

Current Sensing AFM (CSAFM) uses standard AFM Contact Mode, including a special nose cone containing a pre-amp along with an ultra-sharp AFM cantilever coated with a conducting film, to probe the conductivity and topography of the sample. By applying a voltage bias

between the conducting cantilever and sample, a current is generated which is used to construct a conductivity image.

CSAFM is compatible with measurements in air, under controlled environments, and measurements with temperature control. The technique is useful in molecular recognition studies and can be used to spatially resolve electronic and ionic processes across cell membranes. It has proven useful in joint I/V spectroscopy and contact force experiments as well as contact potential studies.

NOTE

CSAFM requires a 10° nose cone with a pre-amp and ultra-sharp, conducting cantilevers.

CAUTION

The CSAFM nose assembly cannot be used for imaging in liquid.

Force Modulation Microscopy (FMM)

In Force Modulation Mode (FMM), the AFM tip is scanned in contact with the sample. As in Contact Mode, a feedback loop is used to maintain a constant cantilever deflection, and an additional, periodic vertical oscillation applied to the tip. The amplitude and phase of cantilever modulation resulting from the cantilever's interaction with the sample varies according to the elastic properties of the sample (Figure 9), with particular sensitivity to elasticity and viscoelasticity.

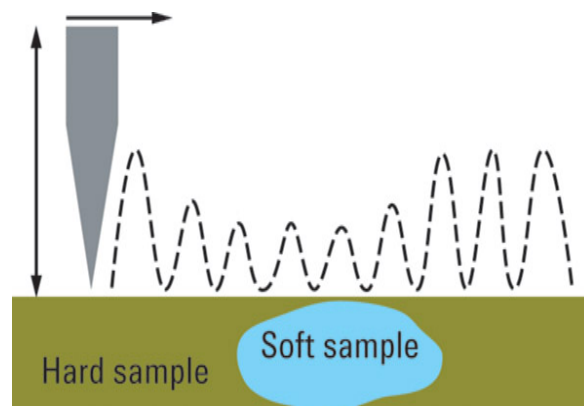


Figure 9 Cantilever response to the applied modulation changes with surface elasticity, as well as other characteristics

NOTE

FMM requires MAC Mode or MAC III.

Lateral Force Microscopy (LFM)

Lateral Force Microscopy (LFM) is a derivative of Contact AFM with the scan direction perpendicular to the long axis of the cantilever. In LFM, the tip is constantly in contact with the sample surface. In addition to its vertical deflection, the cantilever also twists in the scan direction. As a result, along with the near-vertical deflection signal which is usually present during Contact Mode AFM, the detector can also collect a sizeable lateral deflection (Friction) signal from the cantilever's twisting motion. The strength of the lateral deflection signal is related to the friction force between the sample surface and the tip; thus, LFM is sometimes called Friction Force Microscopy.

The LFM signal is highly affected by topographic variations: the rougher the sample surface, the more the topography will affect the friction signal. To differentiate between friction and topography, two images are typically captured side-by-side. One is constructed from the detector signal during the trace (left-to-right tip motion) of each line in the raster scan, and the other is mapped during retrace (right-to-left tip motion). Then one of the two images is inverted and subtracted from the other. This reduces the topographic artifacts in the LFM signal, leaving an image of primarily frictional forces.

Dynamic Lateral Force Microscopy (DLFM)

In Dynamic Lateral Force Microscopy (DLFM), the tip is in contact with the sample, and the cantilever is oscillated parallel to the sample surface (as opposed to perpendicular oscillation in AC Mode). The topography is determined by cantilever deflection, as in contact mode. However, the lateral oscillation is also monitored, such that the amplitude and phase can be imaged, as in standard AAC Mode. DLFM is used in polymer studies as it is very sensitive to changes in surface properties such as friction and adhesion.

NOTE

DLFM requires MAC Mode or MAC III.

Magnetic Force Microscopy (MFM)

Magnetic Force Microscopy (MFM) probes the force between a ferromagnetic tip and a ferromagnetic or paramagnetic sample to image domain structures. The system detects changes in the phase of the cantilever due to interatomic magnetic force that persists for greater tip-sample separation than the van der Waals force.

A standard topography image can be collected for the same scanned area, using AAC in Intermittent Contact mode. The two images can then be displayed side-by-side to highlight any correlation between the magnetic structure and topography.

NOTE

MFM requires AAC, MAC, AAC III or MAC III controller.

Electrostatic Force Microscopy (EFM)

Electrostatic Force Microscopy (EFM) is a qualitative method for examining changes in the intrinsic, or applied, electrostatic field of a sample surface. A voltage bias is applied between the tip and the sample, allowing local static charge domains and charge carrier density to be measured.

The system detects changes in the phase response of the cantilever which are induced by the interaction of the conducting tip and the electrostatic field of the sample surface. EFM images are usually obtained by monitoring the phase change of the cantilever oscillation at the applied frequency.

A standard topography image can be collected for the same scanned area, using AAC (or MAC) in Intermittent Contact Mode. The two images can then be displayed side-by-side to highlight any correlation between the electrostatic response and topography.

NOTE

EFM is an option for the 5500 SPM and requires the additional MAC III controller.

Kelvin Force Microscopy (KFM)

Kelvin Force Microscopy (KFM) is similar to EFM, but with the addition of a feedback loop to maintain a DC tip bias that counteracts

the surface electrostatic force. The output from this feedback loop provides a quantitative analysis of changes in the applied or intrinsic electrostatic field of the sample.

As in EFM mode, KFM uses a conductive tip and either standard AAC or MAC Modes.

NOTE

KFM is an option for the 5500 SPM and requires the additional MAC III controller.

2 Keysight 5500 SPM Components

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The major components for the Keysight 5500 SPM are shown in Figure 10.

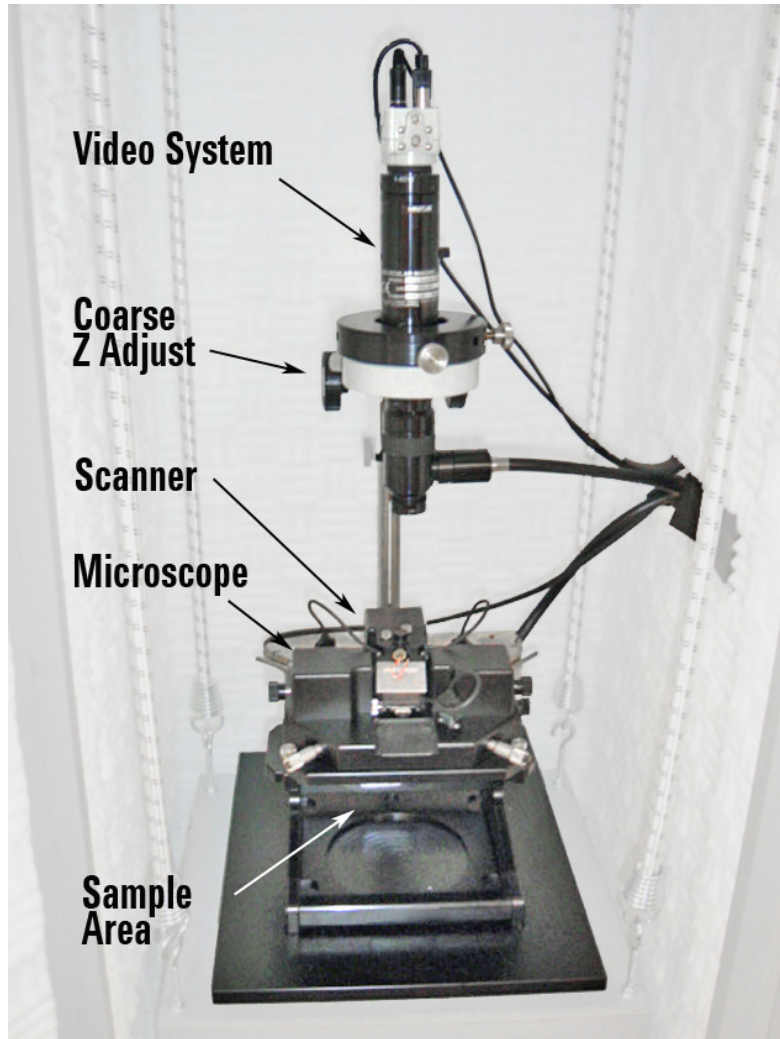


Figure 10 Components of the Keysight 5500 SPM

Microscope

The microscope (Figure 11) includes the hinged support stand, coarse z-axis motors, manual X/Y positioning micrometers, magnetic supports for the sample plates, and interconnections for all electronics. The support stand is hinged to allow easy access to the sample plate area.

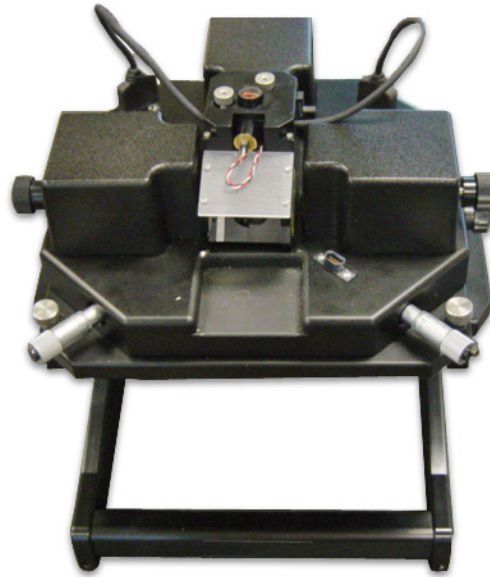


Figure 11 5500 SPM microscope on support stand

Probes

The SPM techniques described in [Chapter 1, Introduction to the Keysight 5500](#), are accomplished using either a wire tip (for STM) or, for AFM imaging, a tip at the free end of a cantilever (a “probe”). STM tips are made by cutting or electrochemical etching Platinum-Iridium or tungsten wire. AFM cantilevers are fabricated from silicon or silicon nitride with an integrated sharp tip at the end.

The selection of probe and tip geometry, cantilever spring constant, and cantilever resonance frequencies will vary depending on application, type of sample surface, imaging environment, and type of image being generated. Tip geometry may be tetrahedral, pyramidal or conical. Tip sharpness, defined by radius of curvature and sidewall angles, greatly affects the resolution available with the probe.

Common cantilever shapes are triangular (V-shaped) and rectangular (beam-shaped). Cantilever spring constants vary from a fraction of N/m (soft) to tens or hundreds of N/m (stiff). Cantilevers for any type of AC Mode imaging (AAC, MAC, etc.) will have resonance frequencies ranging from tens to hundreds of kilohertz.

Cantilevers for particular imaging modes may be coated with reflecting, conductive or magnetic materials.

Nose Assembly

The nose assembly retains the cantilever and enables its motion. A spring clip on the nose assembly secures the probe in place. The nose assembly is held securely in the scanner by an O-ring.

One-Piece Nose Assemblies

The most widely used nose assemblies consist of a single unit which is installed in the scanner (Figure 12). One-piece nose assemblies are available for different modes and may include additional electronics and/or components. For example, the Top MAC nose assembly includes a coil that provides an oscillating magnetic field.

Additionally, nose assemblies are designed to hold the probe at either nine degrees or eight degrees from horizontal. Nine-degree nose assemblies are used for general purpose imaging. Nine and eight-degree nose assemblies may be used for imaging in liquid. A Top MAC nose assembly is available with an eight-degree nose assembly option.

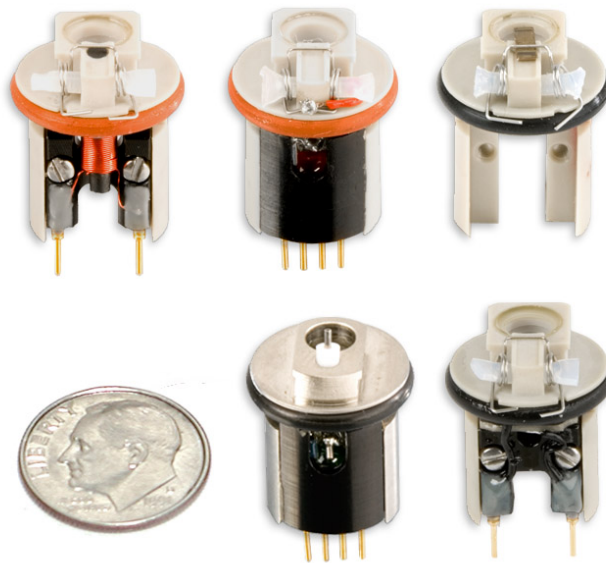


Figure 12 One-piece nose assemblies. Clockwise from upper left: Top MAC, CSAFM, Contact Mode, AAC, STM

CAUTION

The CSAFM nose assembly cannot be used for imaging in liquid.

Two-Piece Nose Assemblies

The all-metal, two-piece nose assembly (Figure 13) was designed to simplify the process of inserting a cantilever. It also helps prevent damage to the scanner during installation of the nose assembly. Currently the two-piece nose assembly is only available for AC Mode/Contact Mode imaging.

CAUTION

The two-piece nose assembly cannot be used for imaging in liquid.

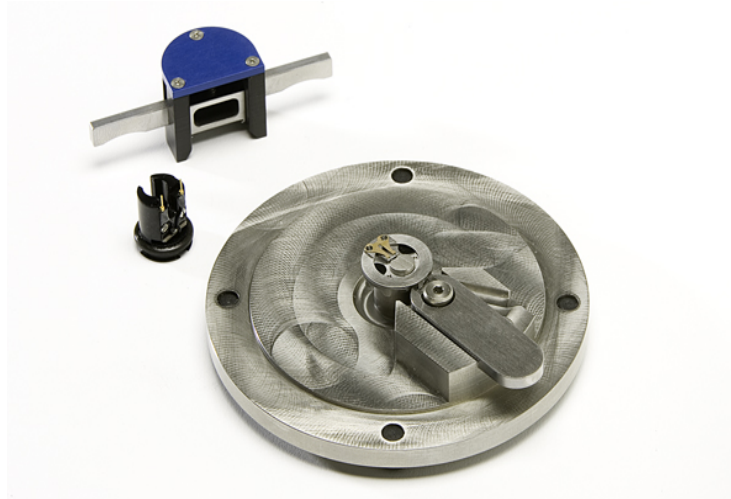


Figure 13 Two-piece nose cone with removal tool and assembly fixture

Scanner

The Keysight 5500 is a tip-scanning system, in which the cantilever sits on a scanner and is moved in raster fashion across the stationary sample. The scanner (Figure 14) includes one or more elements made from piezoceramic material. When an electric field is applied to the piezo elements, they elongate or contract. The motion of the tip in the Z axis, and raster scanning in the X and Y axes, are all achieved by applying high voltages to the scanner's piezo element(s).

Keysight's multi-purpose scanner modules contain the piezo elements, the socket for the nose assembly, mounting for the detector, and interconnections. The scanners are considered "multi-purpose" because nose assemblies can be switched in and out of the scanner for different imaging modes or environments.



Figure 14 A-type Scanner Module

Keysight SPMs use two types of scanners: A and B. A-type scanners are most typically used with the Keysight 5500 SPM (B-type scanners can be used though the video system will not be functional).

A-type scanners that use a 980 nm IR wavelength laser diode are also available for those using inverted microscopes. Additional details for aligning such lasers are in [Chapter 4](#).

There are four A-type scanners:

- The small multi-purpose scanner includes four piezo plates (two each for X and Y motion) and a small piezo tube (for Z motion). The scanner provides scans up to 10 microns square. It is capable of atomic-level resolution imaging.

- The large multi-purpose scanner includes four piezo elements for X and Y and provides scans up to 90 microns square. There are also 2 piezo tubes for Z motion. It provides high resolution and speed for general use applications.
- The large multi-purpose scanner is also available with closed-loop positioning, in which ultra-precise positioning sensors measure displacement in the Z axis only, or X/Y/Z axes. Closed-loop scanning provides superior positioning and more accurate z-position.
- An STM-specific scanner is purpose-built for Scanning Tunneling Microscopy.

Detector

The photodiode detector receives the reflection of the laser spot off the back of the cantilever. The top and bottom halves of the detector monitor the cantilever deflection (the Deflection signal) for AFM imaging, while the two side halves report cantilever twisting (the Friction signal) for lateral force imaging.

The detector mounts in the scanner, typically while the scanner is on the microscope. Two thumbwheels on the detector enable alignment in both directions. There are also four DIP switches to increase or decrease the gain of the signal from the detector.



Figure 15 Detector assembly, top and bottom views

Sample Plates

The Keysight 5500 SPM is designed to allow scanning from above the sample. A variety of sample plates provide mounting options and micro-environments for imaging (Figure 16). The standard sample plate has a magnetic core that will securely hold samples mounted on magnetic backings. Other plates are available for measurement in liquid, temperature controlled imaging, for MAC and other applications. More information for imaging in liquid is in Chapter 12, Liquid Cell.”

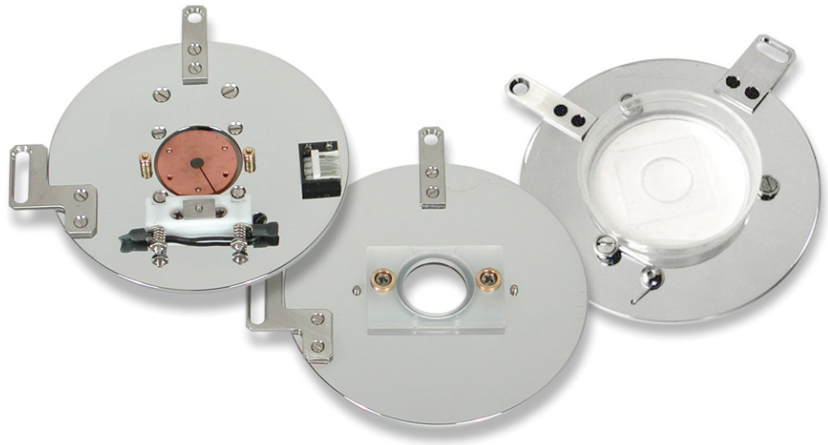


Figure 16 Three sample plates: MAC Mode, liquid imaging, and Petri dish

The microscope stand is equipped with three magnetic posts from which a sample plate is mounted (Figure 17). Micrometers enable manual X/Y positioning with total travel of 5 mm.

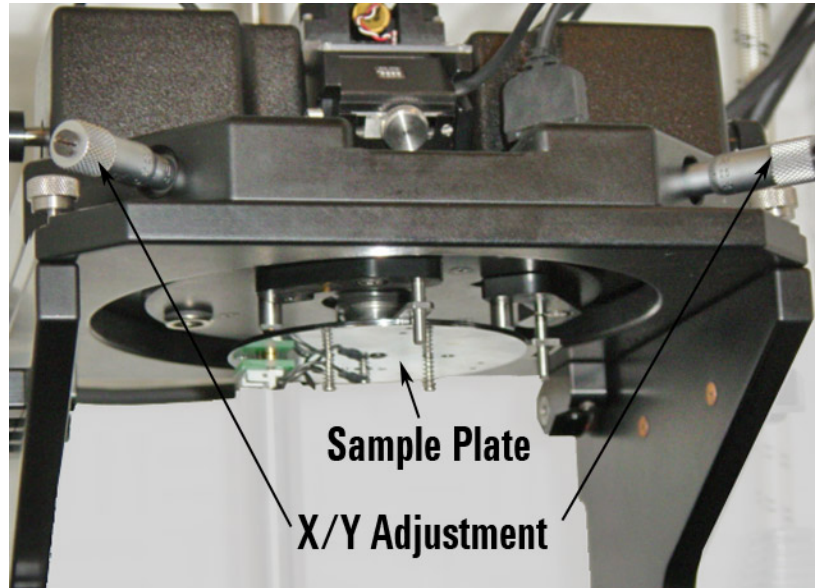


Figure 17 Sample plate on microscope stand

Video System

The video system lets you locate regions of interest and align the laser on the probe tip. It includes a camera and optics on an adjustable stand (Figure 18) along with a separate illumination source (Figure 19). A USB cable connects the camera to the computer.



Figure 18 Video system



Figure 19 Video system illuminator

Head Electronics Box (HEB)

The Head Electronics Box (HEB) (Figure 20) reads the signals from the detector and can display the Sum signal (sum of all four quadrants) and the Deflection or Friction signals. Meter A (upper left) is the Sum signal reading and Meter B (upper right) shows Deflection or Friction (LFM) depending on the state of the switch directly below the meter.

The Open/Close switch is used to move the sample and tip further apart (Open) or closer together (Close).

The HEB also provides an oscillating voltage for AC Mode imaging.



Figure 20 Head Electronics Box

The HEB rear connections are shown in Figure 21:

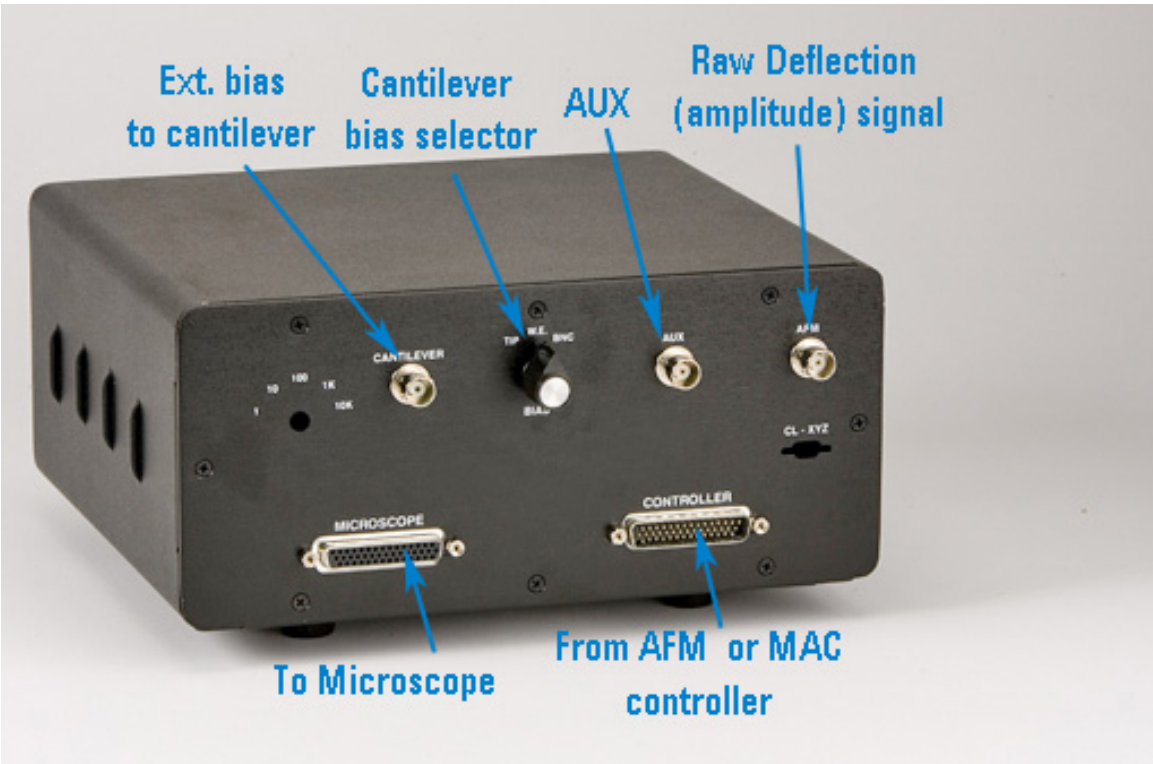


Figure 21 HEB back panel connectors and controls

AFM Controller

The AFM Controller (Figure 22) provides the high voltage to the piezoes and other control functions. Model N9605A is standard; Model N9610A provides optional closed-loop scanning control.



Figure 22 AFM Controller (Model N9610A)

The **Aux In** BNC input is added on to the current signal, which is used in STM and CSAFM. The **External Input** BNC to the Z piezo is limited to ± 10 V and the input is scaled up by the controller to ± 215 V. The **External Sync** BNC is a signal that is sent each time a frame, line, or pixel is completed. This can be used in Custom Spectroscopy or

Scripting. The Sync signal is not accessible with some previous versions of PicoView.

Breakout Box

There is an optional breakout box (N9447A) to access signals from the SPM. This box is shown in Figure 23 and described in Table 1.

“Breakout Box Connections” must be made properly and are shown in Figure 24 on page 50:



Figure 23 Breakout box for AFM controller

Table 1 List of signals and descriptions for AFM controller breakout box

Signal	Description
Error Signal	This is the servo signal from the Head Electronics Box to the controller for AFM/STM. This signal is the photo detector A-B signal with the force setpoint subtracted in AFM mode or the tunneling current in STM mode. If the breakout box is placed between the AC Mode controller and the AFM controller and AC Mode is On , then the signal will be the Amplitude signal from the AC Mode controller. This signal is the Amplitude of the cantilever minus the Amplitude setpoint. This input has a range of ± 10 V.
Friction	This is the LFM signal from the Electronics box to the controller for AFM. This signal is the photo detector C-D signal. If the breakout box is placed between the AC Mode controller and the AFM controller and AC Mode is On , then the signal will be the Phase signal from the AC Mode controller. This input has a range of ± 10 V.
VEC	This is the potential signal from the PicoStat (Potentiostat) board if the microscope has the electrochemistry option. If there is no electrochemistry option, this is a floating input into the AFM controller and can be used as an auxiliary data input channel. This input is displayed by the software with units of volts. This signal is not affected by the presence of the AC Mode controller in the system. This input has a range of ± 10 V.
IEC	This is the current signal from the PicoStat board if the microscope has the electrochemistry option. If there is no electrochemistry option, this is a floating input into the AFM controller and can be used as an auxiliary data input channel. This input is displayed by the software with units of current. This signal is not affected by the presence of the AC Mode controller in the system. This input has a range of ± 10 V.

Current	This is the current signal from the STM or CSAFM pre-amp. This signal is not affected by the AC Mode controller. If there is no pre-amp installed in the scanner, this signal is still driven by a buffer in the HEB so it is only available for use as an auxiliary input if the switch is placed in the IN position and the auxiliary signal would then be fed into the IN BNC only. This input channel is summed into the Aux In BNC on the front of the controller.
Ref Set	This signal is an output from the controller and is the Reference Setpoint DAC output. This signal has a range of ± 10 V and is a 24 bit DAC output.
Bias	This signal is an output from the controller. It is the Sample Bias DAC output or Force Setpoint DAC output (Amplitude Setpoint in AC Mode). This signal has a range of ± 10 V and is a 24 bit DAC output.
+5 V	This is the 5 V power supply output from the controller.
+15 V	This is the +15 V power supply output from the controller.
-15 V	This is the -15 V power supply output from the controller.
Force Setpoint	This signal is an output from the controller and is the Force Setpoint DAC output. This signal has a range of ± 10 V and is a 24 bit DAC output. This signal is the Amplitude Setpoint in AC Mode.
Tip Bias	This signal is an output from the controller and is the Tip Bias DAC output. This signal has a range of ± 10 V and is a 24 bit DAC output.
X HV	This is the +X High Voltage signal used to drive the piezo for scanning in the X direction. This output is ± 215 V and is current limited to 70 mA.
-X HV	This is the -X High Voltage signal used to drive the piezo for scanning in the X direction. This output is ± 215 V and is current limited to 70 mA.
Z HV	This is the Z High Voltage signal used to drive the piezo for scanning in the Z direction. This output is ± 215 V and is current limited to 70 mA.

Y HV	This is the +Y High Voltage signal used to drive the piezo for scanning in the Y direction. This output is ± 215 V and is current limited to 70 mA.
-Y HV	This is the -Y High Voltage signal used to drive the piezo for scanning in the Y direction. This output is ± 215 V and is current limited to 70 mA.

The signals are all passed through the box when the switches are in the OUT position and the signals are all accessible from the IN or OUT BNC's. If a switch is in the IN position, the signal on the OUT BNC is the signal from the source and the connection between the IN BNC and the OUT BNC is broken along with the connection through the box. This allows for some external modification of the signal. The modified signal would then be fed into the system using the IN BNC. The source of the signal is from the Controller for all outputs and from either the microscope electronics or the AC Mode controller for all of the inputs.

Breakout Box Connections

It is important to connect the breakout box correctly as shown in [Figure 24](#).

To access the low voltage ports of the breakout box, connect one end of a low voltage DB-44 cable to the front panel of the AFM controller. Connect the other end of the cable to the N9744A connector labeled **Controller**. Connect one end of a second DB-44 cable (included with the breakout box) to the N9744A connector labeled **Microscope**. Connect the other end of the second cable to the connector labeled **Controller** on the first microscope component in the series configuration. For example, this could be a MAC or AC mode controller, or possibly the Head Electronics Box.

To access the high voltage ports of the breakout box, connect one end of a high voltage DB-9 cable to the front panel of the AFM controller. Connect the other end of the cable to the appropriate 9-pin connector located on the side of the N9744A breakout box. Connect one end of a second DB-9 cable (included with the breakout box) to the second 9-pin

connector on the side of the breakout box. Connect the other end of the second cable to the 9-pin connector on the 5500 microscope base.

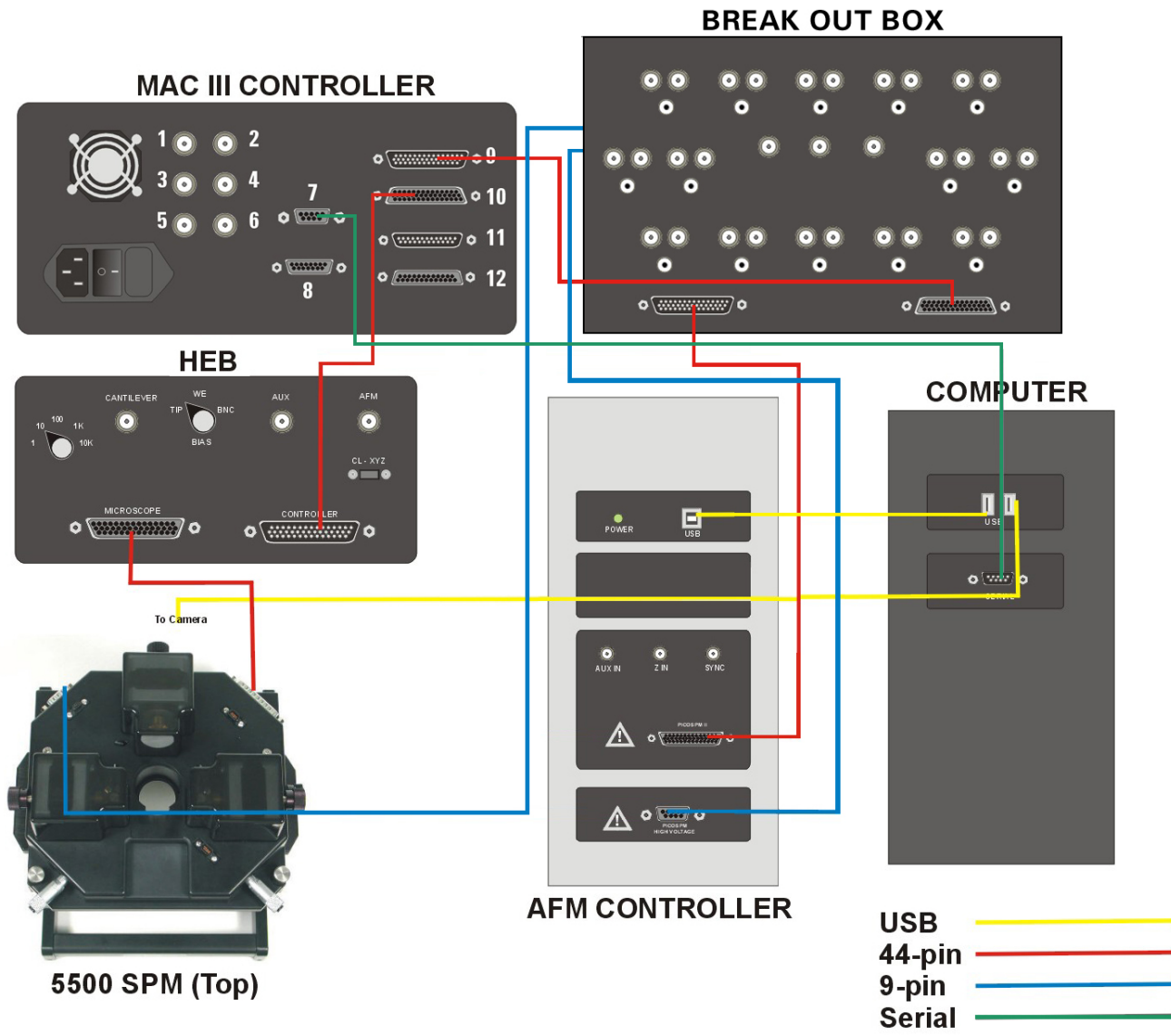


Figure 24 Wiring diagram for breakout box

Vibration Isolation Chamber

The isolation chamber (Figure 25) isolates the 5500 SPM from vibration, air turbulence and acoustic noise which would adversely affect imaging. It also, to an extent, helps control temperature variability.

The chamber provides acoustic isolation for the instrument with multiple layers of sound-damping materials. The vibration isolation system damps incoming vibrations through the use of bungees and suspended mass. The system is designed to be accessed from either the left or right side as both the door and cable ports are reversible.

The enclosure is considered a “mandatory option,” as the improvements it provides for imaging are essential for all but the most stringently controlled environments.

The details for initial assembly are in "[Assembling the Vibration Isolation Chamber](#)" on page 64 of [Chapter 3](#), Setting Up the Keysight 5500 SPM.



Figure 25 Vibration isolation chamber

Software

The Keysight 5500 SPM includes PicoView, a powerful software package for controlling all aspects of alignment, calibration, imaging and more. Integrated in the software are windows for controlling digital cameras and displaying video or still-image output. The system includes post-acquisition PicoImage software for image analysis and data manipulation offline.

To accomplish the steps in the following chapters you will need some familiarity with PicoView. The software steps will be documented briefly in this manual. For more information on the software, please review the online PicoView User Guide. The online User Guide is intended to explain the use of PicoView's screens and controls. You can click the F1 button on your keyboard from most PicoView windows to access this Help file. In most cases, clicking F1 will take you directly to the pertinent topic. There is also a "breadcrumb" trail along the top of the PicoView User Guide window to aid in navigation.

NOTE

The procedures and descriptions in this User Guide are for PicoView 1.12x and higher.

System Options

Many options are available for the Keysight 5500 SPM. As discussed above, probes, nose assemblies and sample plates are available for particular applications. Scanner options include large and small scan ranges, closed-loop scanning, and a dedicated STM scanner. Other options include:

MAC Mode

The MAC Mode options includes the hardware required for MAC mode, which greatly improves imaging in fluid. The options include the MAC controller (Figure 26), and the Top MAC nose assembly, AAC nose assembly and/or MAC sample plate.



Figure 26 Mac Mode Controller

MAC III Mode

MAC III Mode provides the benefits of regular MAC mode, provides three lock-in amplifiers for flexibility, enables EFM and KFM imaging, and provides “Q control” for more precise control of cantilever oscillation.

Liquid Cell

A standard liquid cell is available for AFM and STM. A flow-through liquid cell is also available with connections for tubing. Additional

information is discussed in [Chapter 12, Liquid Cell.](#) A schematic drawing of the liquid cell is shown below in [Figure 27.](#)

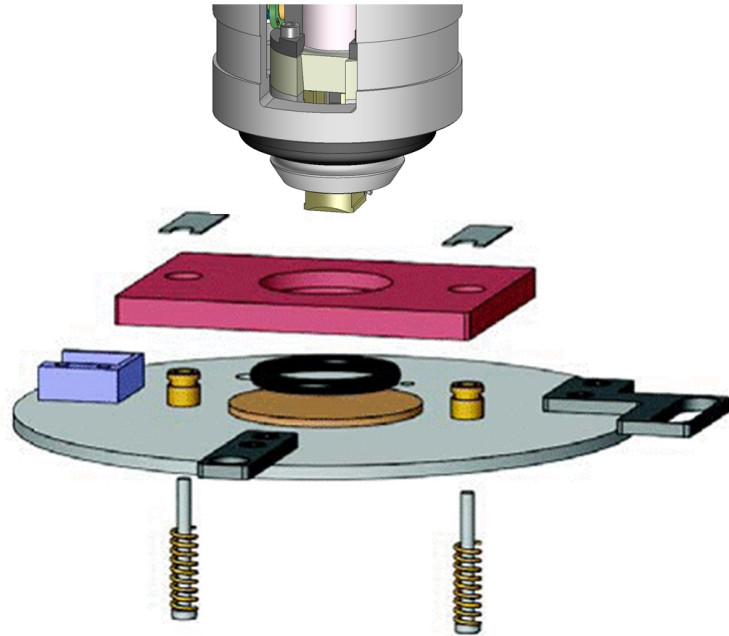


Figure 27 Schematic drawing showing scanner with nose assembly, liquid cell, o-ring, and sample plate

Temperature Control

This option includes low and/or high temperature sample plates, a temperature controller and related hardware for maintaining sample temperature during imaging. Additional information is provided in [Chapter 13.](#)

Thermal K

Thermal K provides a method for accurately determining the force constant of the cantilever for highly accurate force measurements. By measuring the thermal oscillation of the cantilever with no drive signal applied, the cantilever force constant can be determined. The option

includes a separate acquisition card that is installed in an empty slot in the system computer. Additional information is provided in [Chapter 14](#).

Environmental Chamber

The environmental chamber ([Figure 28](#)) allows imaging in controlled atmospheres. Ports and fittings enable gases, liquids and probes to be introduced to the chamber. Additional information is provided in [Chapter 15](#).



Figure 28 Environmental chamber

Glove Box

This small glove box, shown in [Figure 29](#), can be attached directly to the microscope body, offering greater environmental control. Since the sample, piezo, and electronic parts are totally isolated from the imaging environment, it is possible to perform experiments under very reactive

conditions without damaging the system or the sample. Additional information is provided in [Chapter 15](#).



Figure 29 Glove box

Electrochemistry

The electrochemical SPM option includes a low-noise potentiostat/galvanostat for *in-situ* EC-STM and EC-AFM. When combined with temperature control, it is possible to obtain valuable information about electrochemical processes that would otherwise be inaccessible. Additional environmental controls allow imaging with no dissolved oxygen in either aqueous or non-aqueous solutions. Additional information is available in [Chapter 16](#).

PicoTREC

The PicoTREC molecular recognition tool kit ([Figure 30](#)) provides a faster method than force-volume spectroscopy for distinguishing molecular binding events. You can also use PicoTREC to explore

dynamic properties of biological systems by imaging patterns of molecular binding and adhesion on surfaces.



Figure 30 PicoTREC controller

PicoLITH

PicoLITH is an optional package for nanoscale positioning and manipulation, and nanolithography. The PicoLITH option includes its own documentation and is not covered in this manual.

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The Keysight 5500 SPM is typically installed by trained Keysight technical staff. This chapter includes information on the facilities requirements and preparation needed prior to installation. It also offers suggestions on how to handle and re-connect the components should you ever need to relocate the system after installation.

Component and Facility Dimensions

The Keysight 5500 SPM system includes, at minimum, the microscope, computer, Head Electronics Box, and AFM controller. Options purchased with your system may include additional hardware.

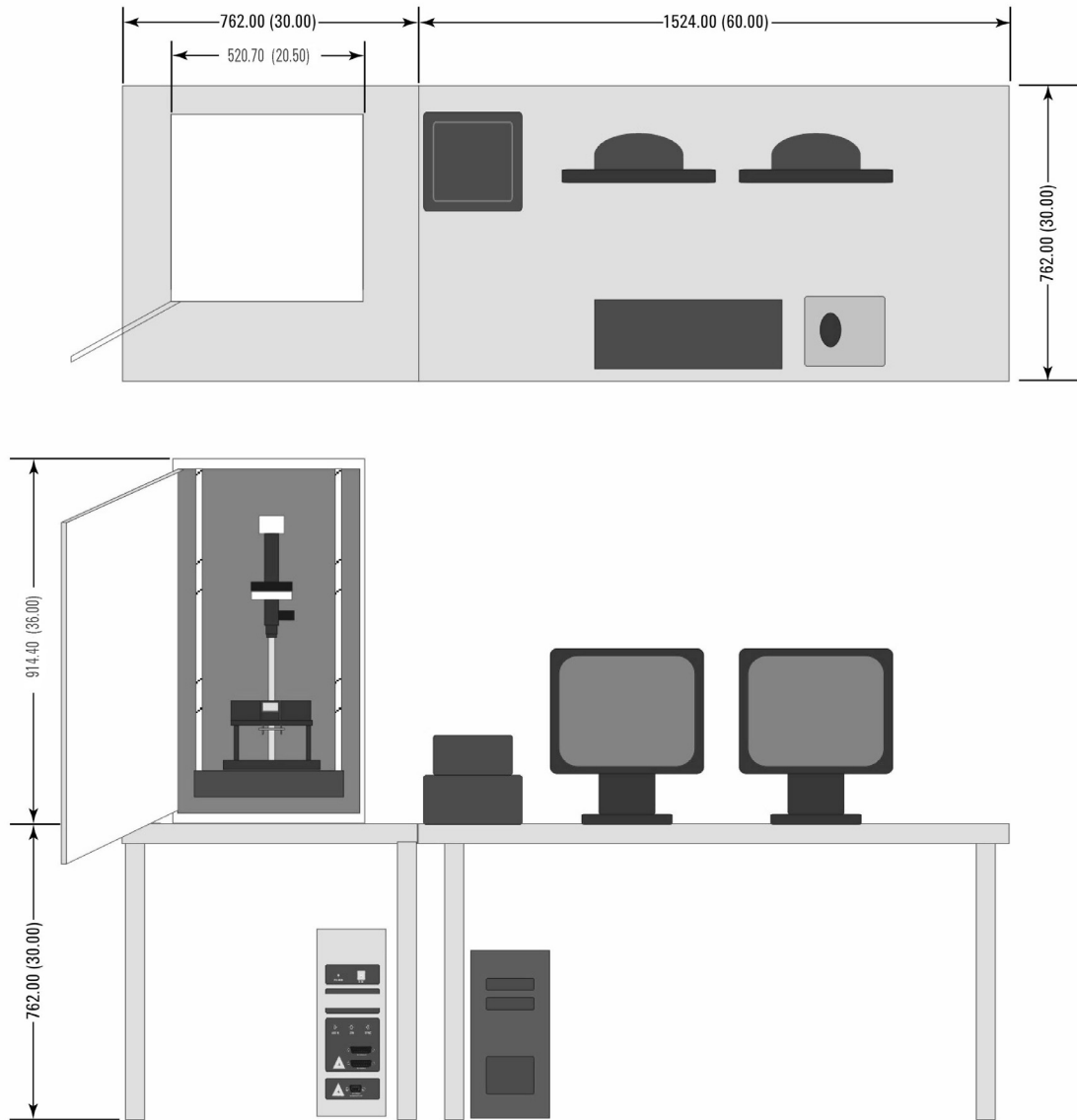
Here are the approximate dimensions of some of the components of the Keysight 5500 SPM:

- 5500 Microscope: 210 mm W x 190 mm H x 160 mm D
(8 in W x 8 in H x 6 in D)

- Head Electronics Box: 203 mm W x 102 mm H x 203 mm D
(8 in W x 4 in H x 8 in D)
- Computer: 203 mm W × 432 mm H × 457 mm D
(8 in W x 17 in H x 18 in D)
- AFM Controller: 178 mm W x 483 mm H x 406 mm D
(7 in W x 19 in H x 16 in D)
- MAC Mode Controller: 254 mm W x 127 mm H x 254 mm D
(10 in W x 5 in H x 10 in D)
- MAC III Controller: 254 mm W x 127 mm H x 254 mm D
(10 in W x 5 in H x 10 in D)
- Vibration Isolation Chamber: 521 mm W × 915 mm H × 603 mm D
(20.5 in W x 36 in H x 23.75 in D)

The most common system configuration includes the 5500 SPM within a vibration isolation chamber, with the controls on a separate table from the rest of the components, as shown in [Figure 31](#) on page 60 (top and front views). Keeping the chamber and microscope on a separate table helps to minimize acoustic coupling from the control station. The isolation chamber and microscope together weight approximately 250

pounds; therefore, a solid table that can easily accommodate 300 pounds is required.



NOTE: DIMENSIONS IN MILLIMETERS (INCHES).

Figure 31 Top and front views of Keysight 5500 SPM suspended inside the isolation chamber, on the left. The control station is on the right.

Facility Requirements

Following these guidelines for preparing the Keysight 5500 SPM facility will ensure a smooth installation, will make using the system more convenient and will improve system performance for the life of the SPM:

- Minimize the acoustic noise level from all possible sources, such as paging speakers, telephone ringer, air conditioner, especially during data acquisition.
- Install the equipment as far away as possible from facility equipment such as air handlers and pumps.
- Keep far away from any source of strong electromagnetic fields.
- Reduce the exposure of the SPM to air flow or dramatic temperature changes. Minimal temperature variation is desirable to minimize thermal drift during measurements and to minimize settling time..
- Use dedicated power outlets with surge protection (strongly recommended).
- Include a set of organized shelves and drawers for system components.
- If gold substrates are frequently used, a hydrogen flame-annealing station is recommended.
- Appropriate water sources should be available for temperature control and biological experiments.

Utilities

The following table summarizes the utility requirements for the Keysight 5500 SPM.

Table 2 Keysight 5500 SPM utility requirements

Configuration	Keysight 5500 SPM
Electrical	1600 W; single phase; 100-120 V or 220-240 VAC; 5 A; 50-60 Hz
Surge protection	Strongly recommended; minimum 7 outlets
Air for isolation chamber	Not required
Internet connection	Recommended

Noise and Facility Specifications

Acoustic Noise

The semiconductor manufacturing industry has developed a standardized set of one-third octave band velocity spectra, called vibration criterion curves (Figure 32), to define acceptable environmental noise. For operation of the Keysight 5500 SPM, facility acoustic noise should be less than 75 dBc. In order to achieve the

specified noise level, the vibration isolation chamber installation surface must meet Criterion D.

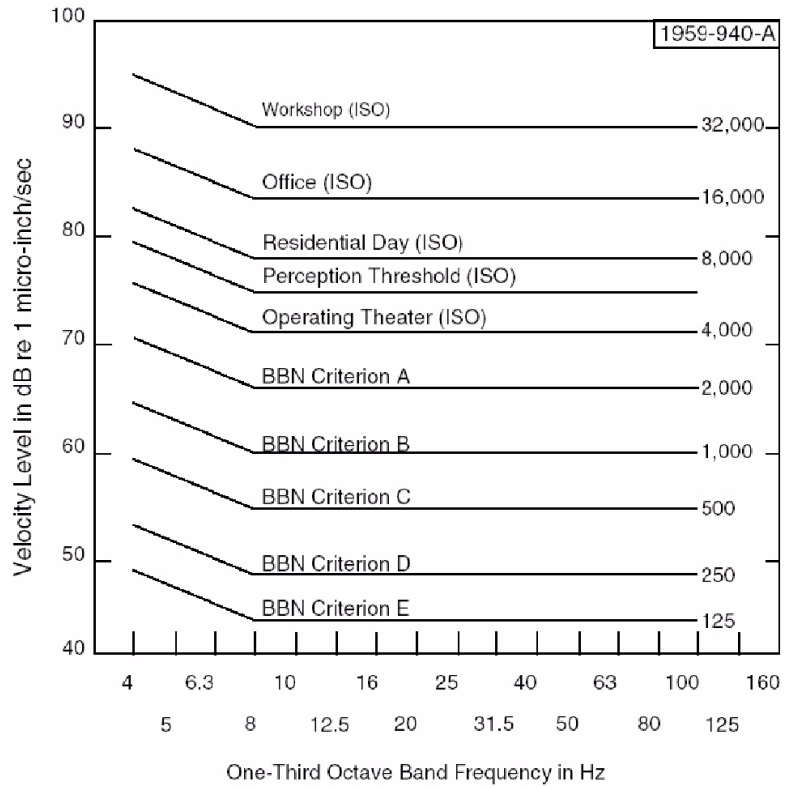


Figure 32 Vibration criterion curves and ISO guidelines

Temperature and Humidity Variation

Changes in temperature and humidity will affect both resolution and repeatability of imaging. Temperature variation should be limited to ± 2 degrees Fahrenheit. Humidity variation should not exceed ± 20 % RH. Locating the instrument away from vents and air handlers will help meet this goal.

Assembling the Vibration Isolation Chamber

Unpack the vibration isolation chamber from the box and remove the packing bag. Save the packing materials for subsequent use.

CAUTION

The chamber is very heavy. Using the lifting handles (see [Figure 33](#)) on the sides of the chamber, two people should move the enclosure to a rigid desk or table. Do not use the chamber door to support any weight.



Figure 33 Vibration isolation chamber with labeled components

Remove the accessory bag from the chamber. This bag contains four adjustable bungee mounts, four bungee cords and a 2.5 mm hex wrench for assembly.

Place the instrument platform, included in a smaller box, on the floor of the chamber.

After unpacking is complete, proceed with assembling the vibration isolation chamber:

- 1 Attach bungee mounts to screws mounted on enclosure ceiling by inserting screw into open end of bungee mount and turning the mounts' pin to the right.
- 2 Attach bungee cords to eyebolts on adjustable bungee mount using bungee cord hook.
- 3 Lift back side of instrument platform and tilt up towards bungee hooks. Attach the back two bungee cord hooks, one at a time, to recessed attachment points as shown in [Figure 34](#) on page 65:
 - a Insert hook into recess hole parallel to the attachment bar.
 - b Turn hook ninety degrees.



Figure 34 Attaching bungee hook to instrument platform

- 4 Repeat [step 3](#) for the front side of the instrument platform.

CAUTION

Do not adjust bungee mounts with SPM instrument on platform.

- 5 Adjust bungee mounts to ensure that the instrument platform is resting level. Turn the standoff using the center pin as shown in [Figure 35](#). Turning the mount to the right will raise the bungee mount; turning the mount to the left will lower the bungee mount.



Figure 35 Adjusting bungee mounts to level the instrument platform

Switching Door Orientation

The door may be hinged on either the left or right side as needed.

- 1 Using the 2.5 mm hex wrench from the accessory bag, detach the 12 fill screws (4 of 12 shown in [Figure 36](#)) from the door and chamber body.



Figure 36 Fill screws on door and chamber body

- 2 Use wrench to detach latch attachment screws (Figure 37). Note orientation of latch parts. Keep screws and latch parts for subsequent reattachment.



Figure 37 Latch with latch attachment screws

- 3 Support weight of enclosure door from below. Use wrench to detach screws which attach the hinge to the enclosure body. Remove door.
- 4 Use wrench to detach hinges from door. Attach hinges to enclosure body on opposite side.
- 5 Put enclosure door back into place and support weight from below. Use wrench to attach hinge to door.
- 6 Use wrench to reattach door latch and to reattach fill screws.

Managing Cables

There are cable ports on both sides of the enclosure. Before installing instrument, select which side of the enclosure the instrument cables should be ported through. If necessary, remove port cover and place in port which will not be used.

- 1 Remove damping foam from cable port to be used. Feed instrument cables out through port.
- 2 Outside of enclosure, compress two pieces of damping foam around cables. Push foam and cables into cable port.
- 3 Adjust as necessary to ensure a good seal around the cables, as shown in Figure 38 on page 68.

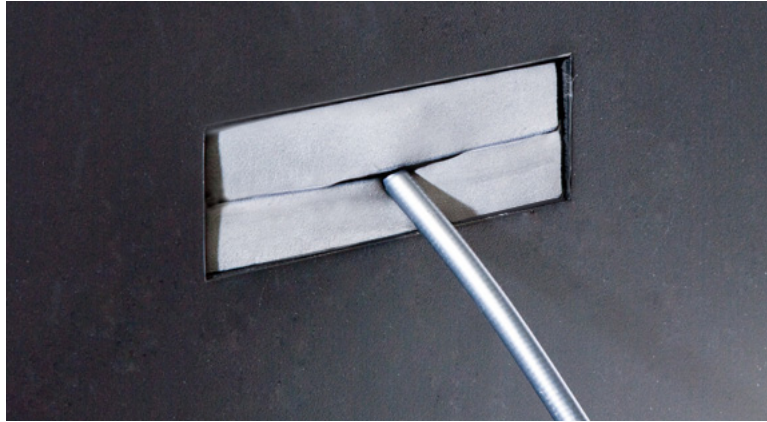


Figure 38 Cables installed with good seal.

Connecting the Components

The cabling for the standard 5500 SPM is shown in [Figure 39](#). Other cabling configurations are included in [Appendix A](#).

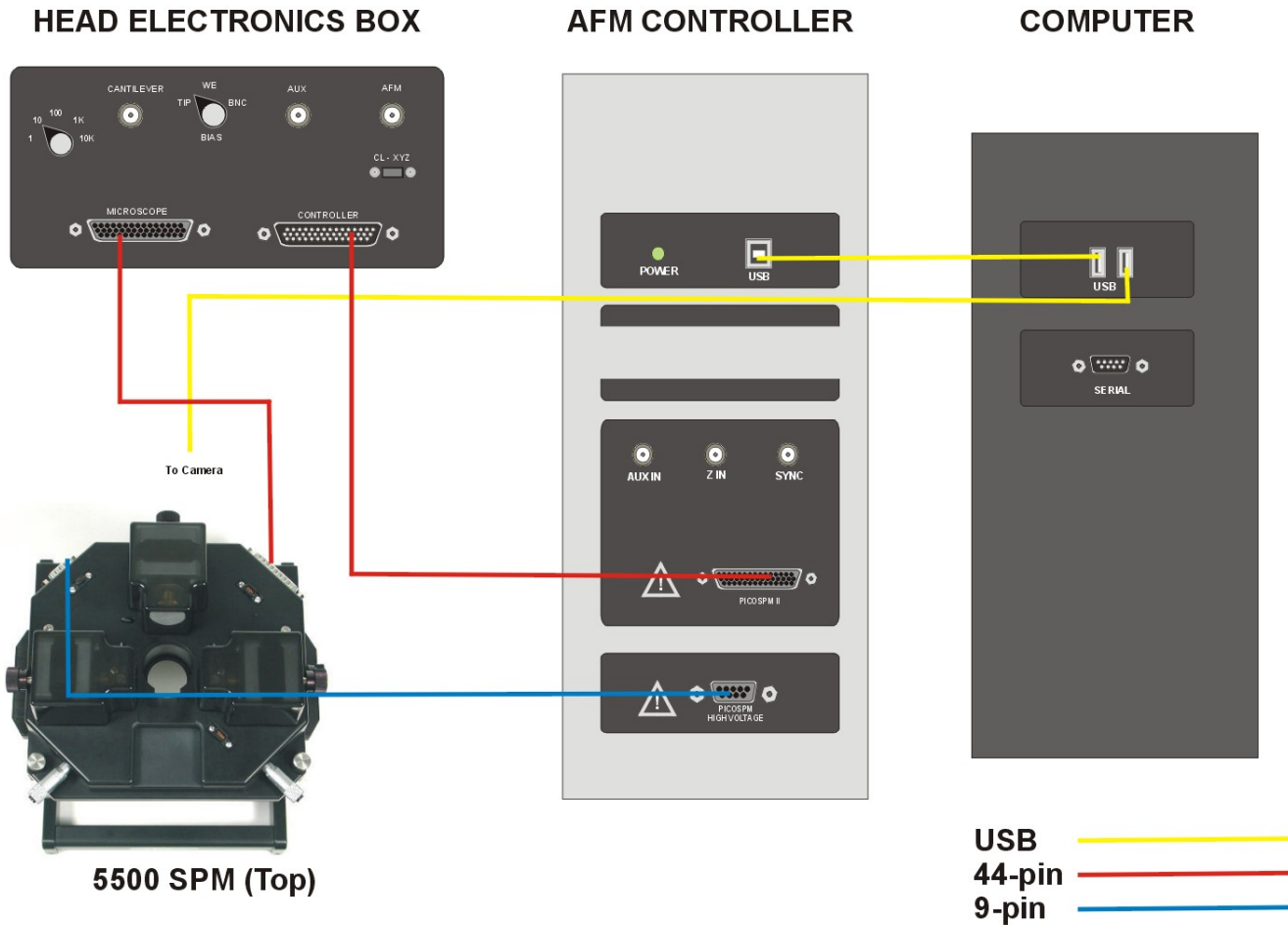


Figure 39 Cabling for basic 5500 SPM configuration

CAUTION

Always make sure that all cables are connected *before* turning on any of the components. Failure to do so can result in damage to equipment.

Guidelines for Moving the System

Should you ever need to relocate the system, here are important guidelines which must be followed to ensure safe operation:

- The new location must meet all of the facility specifications described above.
- Turn off all components before disconnecting cables.
- Disconnect all cables before moving any components.
- Remove the scanner, detector and sample from the 5500 SPM before moving the microscope base.
- Remove the microscope base from the vibration isolation chamber and transport both separately.
- Follow the cabling diagrams exactly, being sure to connect all cables before powering up the components.

Care and Handling of the Probes and Scanner

Probes

Always store probes at room temperature in their protective cases.

Handle probes gently with tweezers, following the procedures described earlier in this chapter.

If a probe is dropped it may very well be damaged. You can check whether the cantilever is intact by viewing it through a magnifier.

If you are using more than one type of probe, be sure to store them separately in well-marked cases to avoid confusion.

Nose Assembly

Put nose assemblies in the specially tailored scanner container box and store in a clean, dry location where they will not be subject to excessive humidity, temperature changes or contact.

Dirt, grease or spots on the glass window of the nose assembly can interfere with the optical path of the laser. Regularly clean the window with cotton or a soft tissue (dry, wetted with water, or with ethanol).

The glass is glued to the nose cone with chemically resistive epoxy, so if the window breaks there is no easy way to replace it and the entire nose assembly will likely need to be replaced.

Only remove the nose assembly from the scanner using the Nose Assembly Removal Tool, with the scanner placed upright in its fixture.

Do NOT use the removal tool to install the nose assembly.

Two-Piece Nose Cone Cleaning

The two-piece nose cone is not to be used in liquid because it does not have a glass window to prevent liquid from getting to the scanner. After it is removed from a scanner, the two-piece nose cone may be cleaned with a low oxidizing organic solvent such as ethyl alcohol.

Scanner

Between uses, remove the scanner from the microscope and store it on its fixture or in the storage case, in a location where it will not be subject

to excessive humidity, temperature changes or contact. Keysight recommends that scanners be stored in a desiccator.

The scanner contains very brittle and fragile piezoelectric ceramics. Applying excessive lateral force while exchanging nose assemblies, or dropping the scanner even a short distance onto a hard surface, will damage the scanner. If the nose assembly housing becomes loose or can be wiggled when in place, contact Keysight support for assistance.

Cracked or broken piezoelectrodes will result in abnormal imaging. Damage to the scanner such as those described above are NOT covered by the standard warranty.

4 Preparing for Imaging

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The Keysight 5500 SPM is capable of imaging in many different modes. Several steps of the imaging process are similar or identical, however, for all modes. This chapter will cover the steps that are common to most imaging procedures.

Setting Up the Scanner Assembly

As mentioned earlier, the Keysight 5500 SPM is a tip-scanning system, in which the probe is raster-scanned across the stationary sample. When an electric field is applied to the scanner's piezo elements, they elongate or contract, depending on the direction of the field. The Z-motion of the tip is achieved by elongation or contraction of the piezo element in the scanner. X/Y raster scanning is achieved by applying alternating

voltages to opposite piezo elements in the scanner so that one element elongates and the other contracts.

CAUTION

The thickness of the piezo elements determines how much they will expand or contract per applied unit voltage. They are necessarily thin to provide scanning resolution. **If dropped, the scanner's piezo elements WILL break.** Cracked or broken piezoelectrodes will result in abnormal imaging. Proper handling is essential to preserve the long expected life of your multi-purpose scanner.

The scanner mounting fixture supplied with your system is designed to keep the scanner and its components safe during handling (Figure 40). The main cutout safely holds the scanner, while the smaller cutout safely holds a nose assembly. A magnetic disk keeps additional tools close at hand.

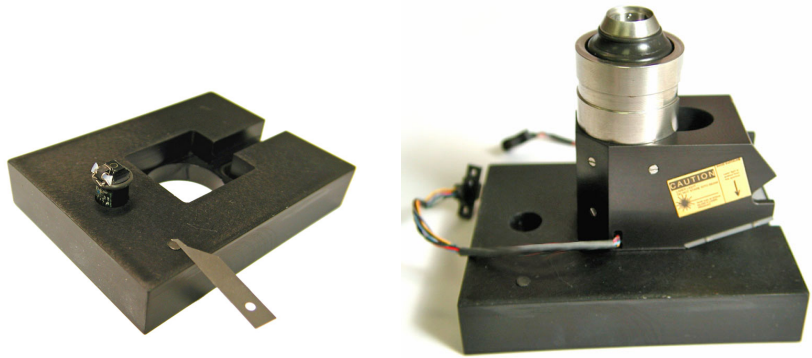


Figure 40 Scanner mounting fixture with nose assembly and spring key; scanner in mounting fixture

The next sections will describe how to safely handle the scanner components for long life and excellent imaging.

One-Piece Nose Assembly

Inserting the One-Piece Nose Assembly

The nose assembly is held in the scanner by an O-ring around its circumference. To insert a nose assembly, first place the scanner in the scanner mounting fixture (Figure 40). Place the nose assembly in the socket on top of the scanner, aligning its contact pins if applicable.

CAUTION

Ensure that the ends of the metal spring for retaining the cantilever are not caught in between the nose cone and the socket.

Applying even, steady, vertical pressure at the edges of the nose assembly, seat it into the socket, as shown in [Figure 41](#).



Figure 41 Apply even, vertical pressure at the edges to insert the nose assembly (as shown on left). O-ring should be fully inside socket (as shown on right).

CAUTION

Push evenly and straight down when inserting the nose assembly. Small off-axis forces will create LARGE torques about the anchor point for the piezoes, where most breakage occurs.

Do NOT push down on the top of the nose assembly as this will damage the spring clip and/or glass window.

Removing the One-Piece Nose Assembly

A removal tool is included with your system to limit damaging, lateral forces on the scanner while removing the nose assembly. The following is the only acceptable procedure for removing the nose assembly:

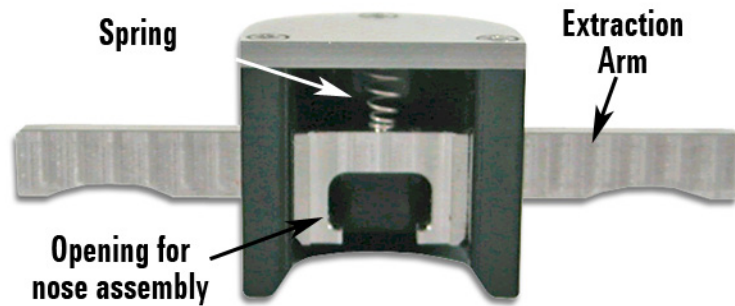


Figure 42 Nose assembly removal tool

- 1 Place the scanner in the scanner mounting fixture.
- 2 Carefully slide the removal tool onto the nose assembly, ensuring that the opening seats on both sides of the nose.
- 3 Position your thumb on the flat surface of the removal tool and your fingers on BOTH sides of the extraction arm.
- 4 Gently pull up with your fingers while pushing down with your thumb (Figure 43).

- 5 Once the nose assembly is clear of the scanner you can remove it from the tool.



Figure 43 Using the nose assembly removal tool

CAUTION

Do not use the nose removal tool to insert a nose assembly. It is not designed for this purpose.

CAUTION

DO NOT use tweezers to remove the nose assembly (Figure 44). Tweezers can create a pivot point to lever the nose out of the scanner, placing large lateral forces on the piezo assembly. The nose assembly removal tool is the only acceptable method for extracting the nose from the scanner.

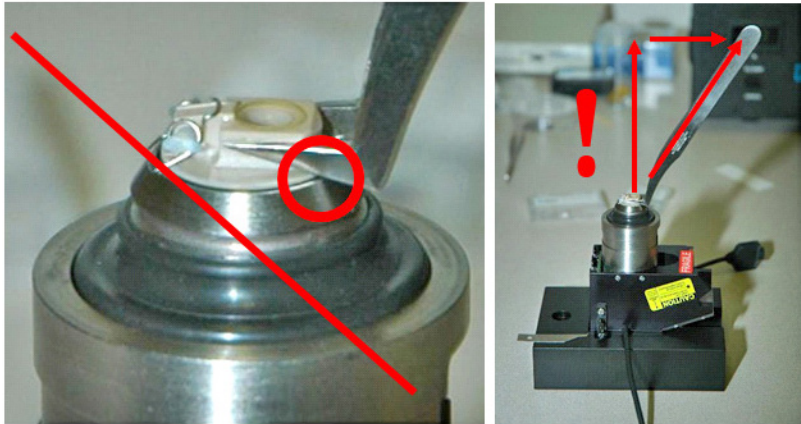


Figure 44 Do not use tweezers to remove a nose assembly. Doing so can place damaging lateral forces on the scanner.

Inserting a Probe in the One-Piece Nose Assembly

Keysight nose assemblies are designed with a spring and guides to retain the probe in the proper position for imaging. A spring key (Figure 45) is included with the system to let you safely hold back the spring while inserting the probe. Figure 46 shows a properly positioned



probe.

Figure 45 Spring key

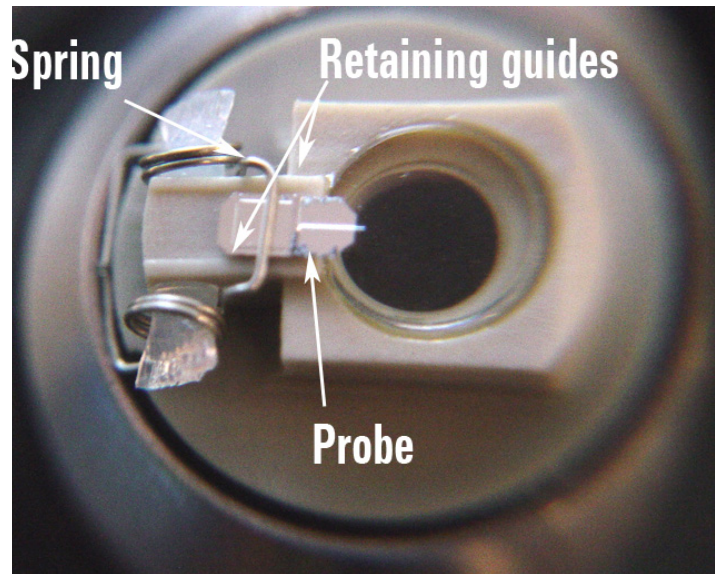


Figure 46 Probe properly situated on AFM nose assembly

AFM probe tips are extremely delicate and can break when dropped even a short distance. The following instructions include several helpful tips that will simplify the process of inserting a probe in the nose assembly:

- 1 Mount the nose assembly in the scanner.
- 2 Place the scanner into the scanner mounting fixture.
- 3 Grasp the tweezers in the orientation shown in [Figure 47](#).
- 4 Gently grasp the probe from its sides, applying just enough pressure to secure it in the tweezers. It is often easier to take the probe from the case, as demonstrated in [Figure 47](#), than to try to grasp the probe with the case sitting on the desk or table. This method allows the probe to be held at an angle, making it easier to insert it into the nose assembly.

CAUTION

Ensure that the ends of the metal spring for retaining the cantilever are not caught in between the nose cone and the socket.



Figure 47 Holding the tweezers as shown, remove a probe from the protective case

- 5 With your free hand, use the spring key to rock back the retainer spring (**Figure 48**).
- 6 Place the probe between the guides such that a little more than half of the probe extends over the lens (placement will vary depending on the type and shape of the probe). **Figure 48** shows this process. The final probe position should be as shown in **Figure 46** on page 79.



Figure 48 Hold back the retaining spring while placing the probe

- 7 Gently lower the spring clip to hold the probe in place.

CAUTION

The retainer spring can snap back with enough force to damage the probe, so be sure to release the spring slowly and gently.

Two-Piece Nose Assembly

The two-piece nose assembly was designed to simplify the process of inserting a probe through the use of an assembly fixture (Figure 49). The nose assembly consists of a body, which inserts into the scanner, and a flat, stainless steel disk which holds the cantilever. The disk is held to the body magnetically and can be separated by holding the disk at its edges and gently pulling it from the body. The proper attachment of the disk is ensured by aligning the three positioning guides on the body of the nose cone with the tabs on the disk.



Figure 49 Two-piece nose assembly, removal tool and assembly fixture. The nose assembly disk is shown on the fixture; the body is shown to its left.

Inserting the Body of the Two-Piece Nose Assembly

As with the one-piece nose assembly, the body of the two-piece assembly is held in the scanner by an O-ring. To insert the body, first place the scanner in the scanner mounting fixture (Figure 40 on

page 74). Place the body in the socket on top of the scanner, aligning its contact pins.

Applying even, steady, vertical pressure with your fingers to seat the body into its socket.

CAUTION

It is essential to push evenly and straight down when inserting the nose assembly. Small off-axis forces will create LARGE torques about the anchor point for the piezoes, where most breakage occurs.

Removing the Body of the Two-Piece Nose Assembly

As with the one-piece nose assembly, a tool (Figure 50) is included to remove the body of the two-piece assembly from the scanner. The tool limits damaging, lateral forces on the scanner during the removal process. The following is the only acceptable procedure for removing the nose assembly body:



Figure 50 Two-piece nose assembly removal tool

- 1 Place the scanner in the scanner mounting fixture.
- 2 Remove the nose assembly disk from the body by gently pulling it up from its edges.
- 3 Carefully slide the removal tool onto the nose assembly, ensuring that the opening seats on both sides.
- 4 Position your thumb on the flat surface of the removal tool and your fingers on BOTH sides of the extraction arm.
- 5 Gently pull up with your fingers while pushing down the your thumb.

Inserting a Probe in the Two-Piece Nose Assembly

AFM probe tips are extremely delicate and can break when dropped. Follow these instructions to safely insert a probe in the nose assembly:

- 1 Place the nose assembly disk on the assembly fixture, as shown in [Figure 51](#). Make sure that it aligns with the center disk and two small alignment pins.

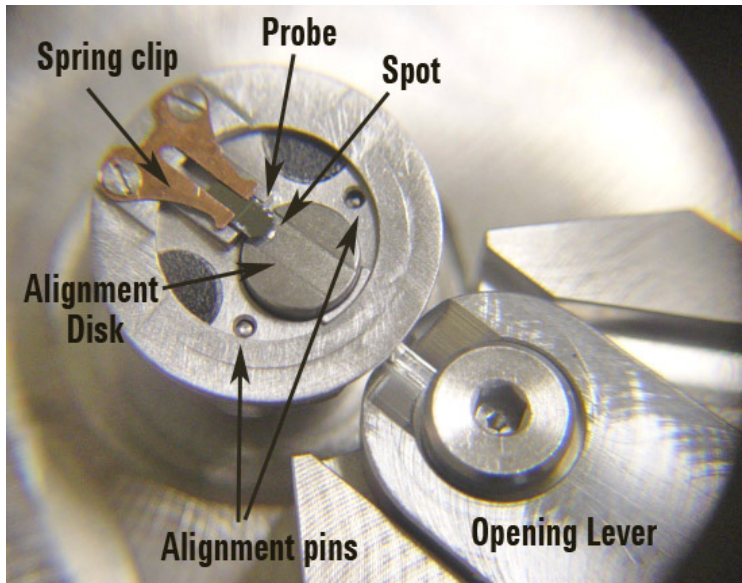


Figure 51 Two-piece nose assembly disk on fixture

- 2 Using tweezers, gently grasp a probe from its sides, applying just enough pressure to secure it in the tweezers.
- 3 Move the assembly fixture lever to the right, which will slightly separate the nose assembly disk ([Figure 52](#)).

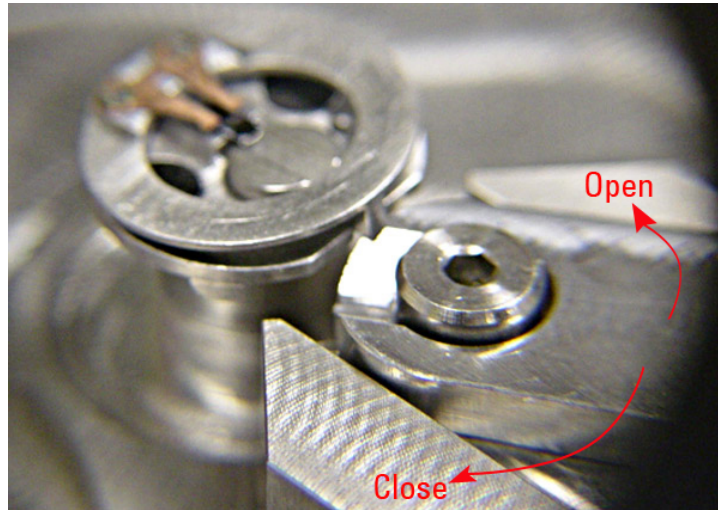


Figure 52 Move the lever to open the nose assembly disk.

- 4 Place the probe under the copper-colored spring clip on the nose assembly disk. Use the alignment guides in the fixture to help locate the probe laterally.
- 5 A small alignment spot on the fixture ([Figure 51](#) on page 83) indicates the proper location for the cantilever tip. Place the probe such that the tip is close as possible to this spot.
- 6 Move the lever to the left to close the nose assembly disk.
- 7 Use the tweezers to finely adjust the probe such that the cantilever is aligned over the alignment spot. Only grasp the probe from the sides to avoid damaging the cantilever.
- 8 Grasping the nose assembly disk from the edges, remove it from the fixture and align it on the nose assembly body already in the scanner.

Inserting the Scanner and Connecting Cables

At this point you should have the probe, nose assembly and scanner all assembled into one unit.

- 1 Make sure there is adequate clearance below the scanner socket in the middle of the microscope.
- 2 Place the scanner assembly into the scanner socket, with the scanner's frosted screen facing up and forward ([Figure 53](#)).

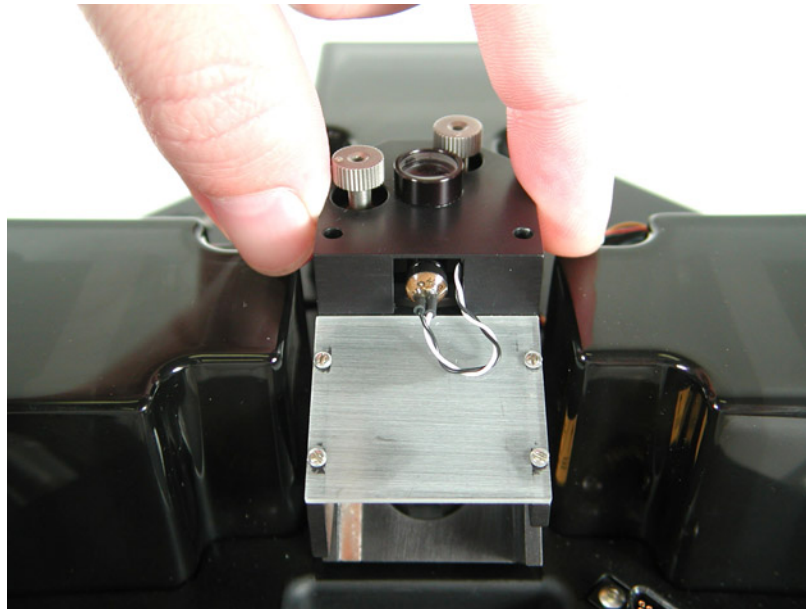


Figure 53 Placing scanner assembly into microscope

- 3 Finger-tighten the knob on the right side of the microscope to lock the scanner in position.
- 4 Attach the high voltage (red) and low voltage (blue) cables on either side of the scanner to the sockets on the microscope base. The cables are color coded to avoid confusion. If you are using a closed-loop scanner, connect its third cable to the C/L socket on the rear of the Head Electronics Box.

CAUTION

To remove the scanner, turn off all power THEN disconnect cables.

Aligning the Laser

The next step is to ensure that the scanner's laser spot is aligned to reflect off of the cantilever. Several methods can be used to do so.

In most cases, particularly with highly reflective samples, you can use the 5500 SPM's video system to focus on the cantilever and align the laser spot (Figure 54). If the sample is not reflective, use a piece of gold film on mica or other reflective sample.

The laser spot will be visible in the video image until it crosses the cantilever, so you can use a similar procedure to the paper method below. See "Using the Video System" later in this chapter.

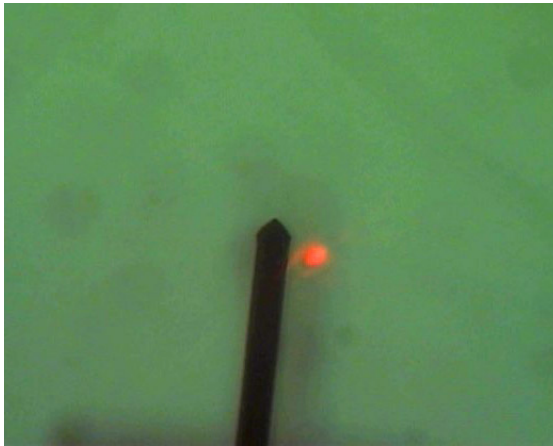


Figure 54 Using video system to align laser

Figure 55 shows how the position of the laser with respect to the cantilever affects the laser reflection to the detector. Due to the variation

of cantilever types and vendors, the position of the laser on the cantilever needs to be optimized per tip.

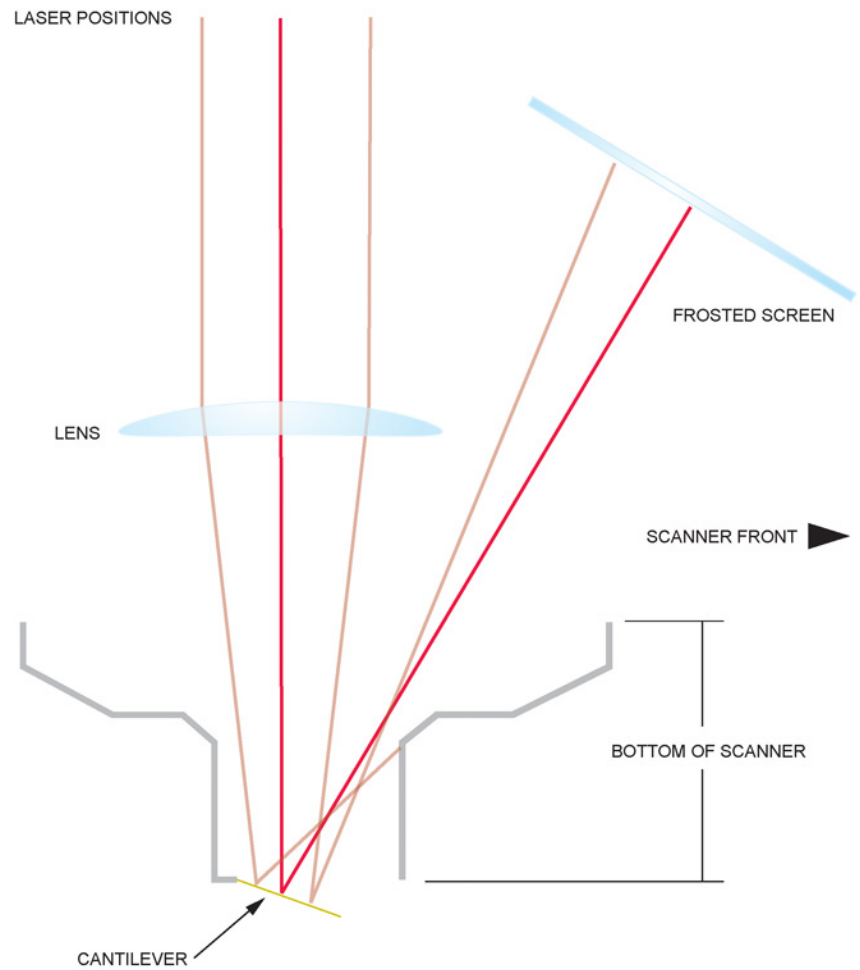


Figure 55 Laser alignment

Note that the IR sensor card should be used for coarse positioning of the laser if using the 980 nm IR scanners.



Figure 56 Coarse laser alignment for IR scanners using sensor card

Another method is to place a white card or piece of paper under the scanner to make the laser spot visible. By moving the laser you can then align it on the probe—when this happens the probe will block the laser spot, and the spot will no longer be visible on the paper.

- 1 Place a white piece of paper or business card on the table below the microscope. If using a 980 nm IR scanner, use an IR sensor card in

place of a business card (see [Figure 56](#)). The operational range of the sensor card is 700 nm to 1400 nm.

- 2 Turn on the Head Electronics Box, which will activate the laser. You should be able to see the red laser spot on the paper ([Figure 57](#)).

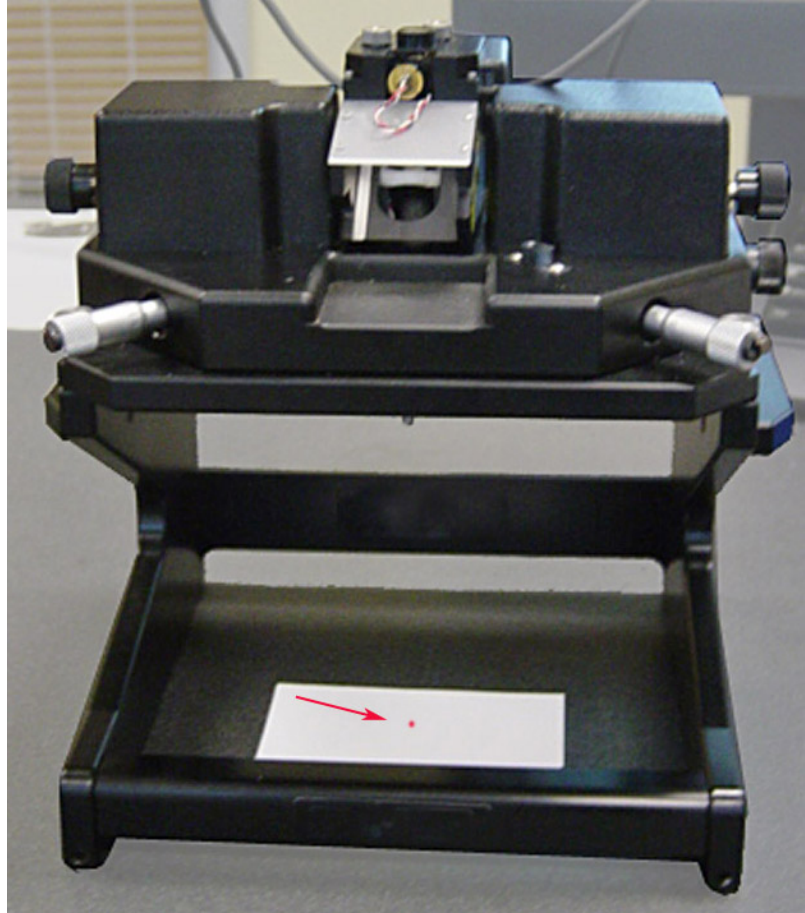


Figure 57 Aligning laser spot over white paper.

The laser alignment knobs are located on the top of the scanner ([Figure 58](#)). The front-to-back knob moves the laser spot toward the

cantilever tip (counterclockwise) or away from it. The left-to-right knob adjusts the lateral position.

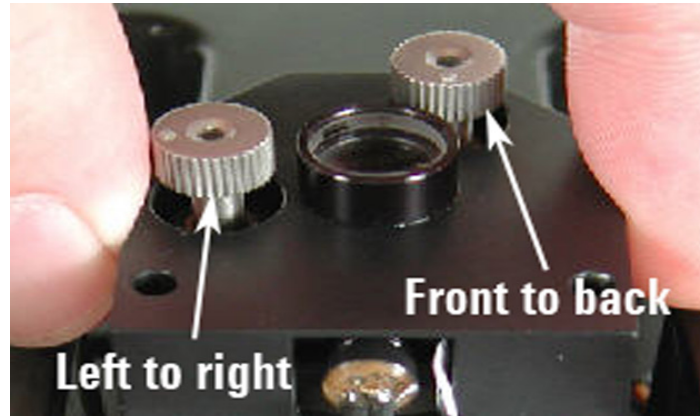


Figure 58 Use the scanner knobs to position the laser spot

- 3 Rotate the front-to-back knob clockwise to move the laser spot onto the cantilever chip (Diagram B in [Figure 59](#)). When the laser reaches the chip it will be blocked and will no longer be visible on the paper. You should only need to turn the knob a few rotations.

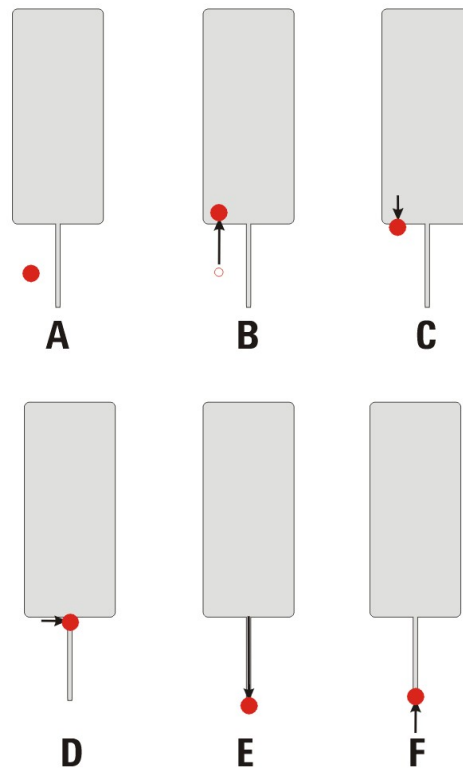


Figure 59 Steps to aligning laser on cantilever beam tip

- 4 Rotate the front-to-back knob counterclockwise until the spot just reappears on the paper. The spot is now at the edge of the chip (Diagram C in [Figure 59](#)).
- 5 Rotate the left-to-right knob to position the laser on the cantilever (Diagram D in [Figure 59](#)). As the laser passes over the cantilever it will disappear and reappear in rapid succession. You should now see the laser spot on the scanner's frosted glass.
- 6 Turn the front-to-back knob counterclockwise to move the spot down the cantilever, toward the tip until the spot on the frosted glass disappears (and the spot reappears on the paper) (Diagram E in [Figure 59](#)).
- 7 Turn the front-to-back knob clockwise slightly to position the laser just on the cantilever tip (Diagram F in [Figure 59](#)). The spot will reappear on the ground glass.

The process is similar for triangular-shaped cantilevers, with the exception that the laser will be obscured twice as it moves left to right (over the two beams). The process is shown in [Figure 60](#).

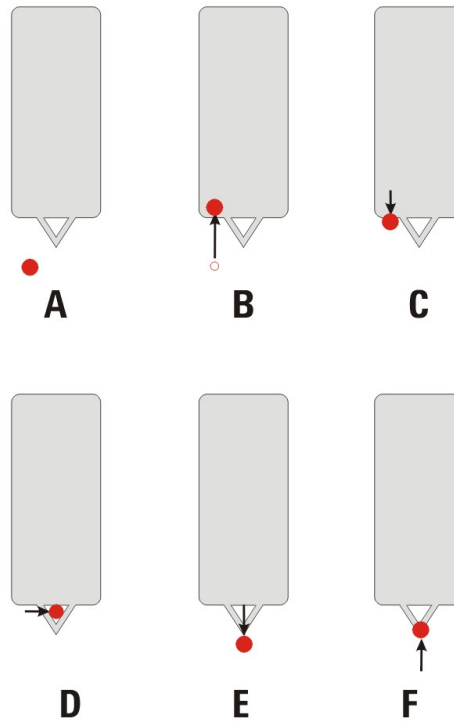


Figure 60 Steps to aligning the laser on triangular cantilevers

A potential error during the alignment process is to turn either of the positioning controls too far in the wrong direction and to thereby lose the laser spot altogether. [Figure 61](#) shows the positioning controls when they are well out of alignment. The left-to-right knob will be visibly tilted when the lateral alignment is far out, and the laser housing will be moved to one side when the front-to-back alignment is out. The easiest way to recover is to roughly center both controls again, moving the laser

back to the center of its travel in both directions. Doing so should make the laser spot reappear.

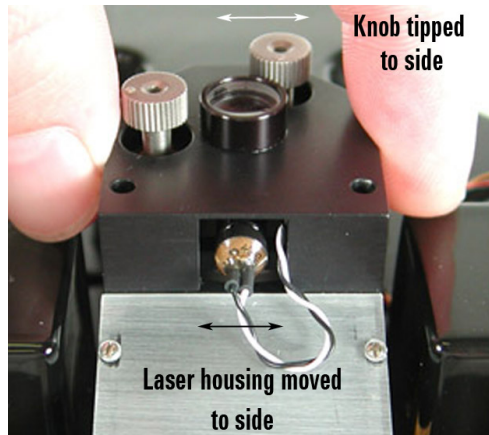


Figure 61 Laser alignment control when far out of alignment

Inserting and Aligning the Detector

The photodiode detector records changes in the position of the laser spot as the cantilever passes over the sample surface. As shown in [Figure 62](#), the detector senses the laser's movement between its four quadrants, reporting the AFM (vertical deflection), LFM (lateral, or friction), and SUM signals.

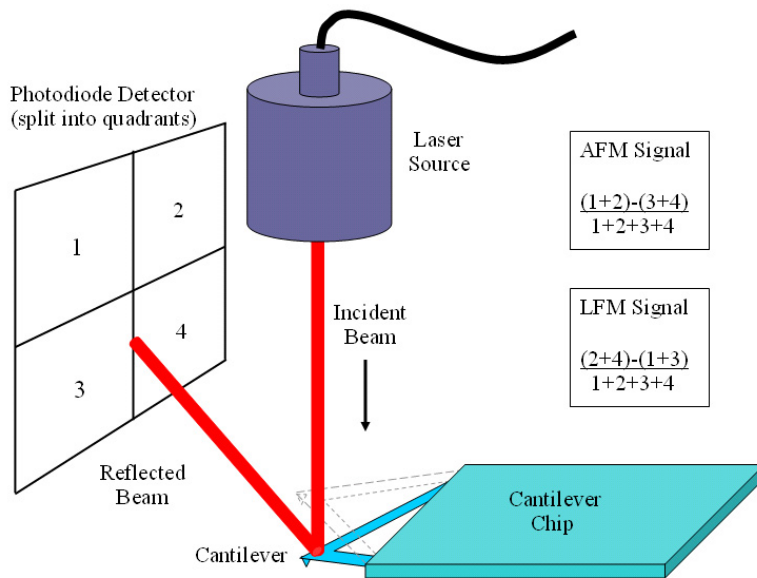


Figure 62 Photodiode detector operation

To install the photodiode detector, insert it into the scanner until it stops ([Figure 63](#)). Plug the detector cable into the Detector socket on the microscope base. You can install the detector into the scanner before or

after installing the scanner in the microscope base, or after you align the laser.

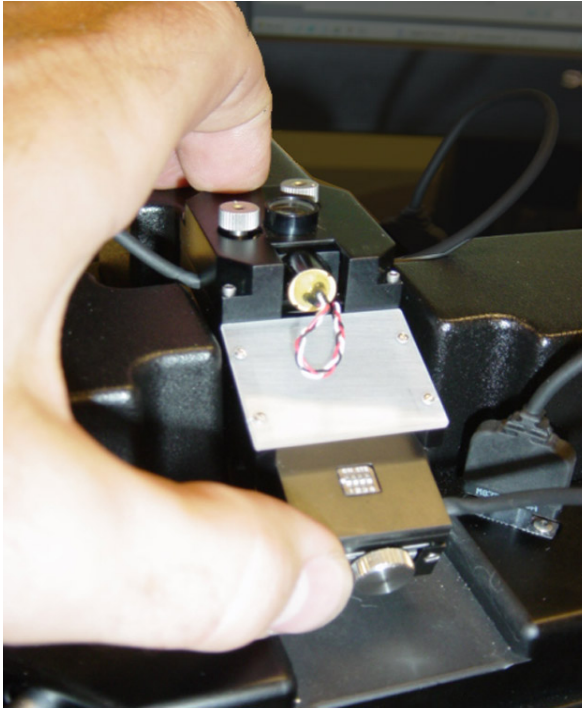


Figure 63 Inserting the detector module into the scanner

The Gain Switches on the detector determine whether the laser signal is amplified before going to the rest of the electronics. **On** (away from the adjustment wheels) means no amplification, **Off** means the signal is amplified. Each switch represents one of the four quadrants in the photodetector. All switches should be either up for normal operation, or down to increase the signal for poorly reflective cantilevers.

Detector alignment is completed through the PicoView software:

- 1 Launch PicoView. The Laser Alignment window (as well as other windows) will open, displaying the position of the laser spot on the

photodiode detector (Figure 64). You can also click the **Laser Alignment** toolbar button to open the window.

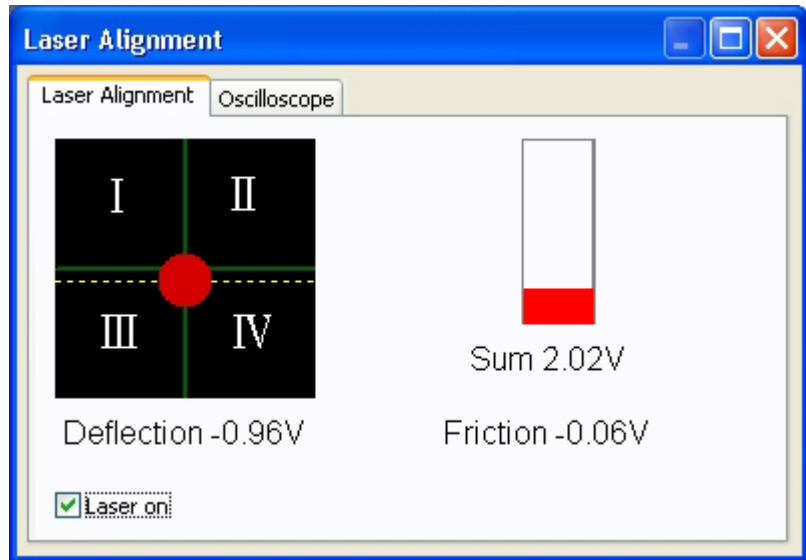


Figure 64 Laser Alignment window in PicoView

The meter on the right of the Laser Alignment window shows the Sum of all four quadrants. The Deflection signal is the difference between the top and bottom halves divided by the Sum. The Friction signal is the difference between the left and right halves divided by the Sum. The laser On and Off can be controlled through the software by selecting or clearing, respectively, the **Laser on** check box.

NOTE

These signals can also be seen on the Head Electronics Box where Meter A is the Sum signal reading and Meter B shows Deflection and Friction (LFM) depending on the state of the switch directly below the meter.

- 2 Use the knobs on the detector to move the laser spot to the center of the four quadrants. The front (deflection) knob moves the spot up (clockwise) or down (counterclockwise). The left (friction) knob moves the spot to the left (clockwise) or to the right (counterclockwise).
- 3 If the laser spot is off the detector, the SUM signal will be close to 0 V and the solid laser spot in the Laser Alignment window will be displayed as an unfilled circle. Take the detector out of the scanner and adjust the position of the detector using the laser spot on the frosted screen on the scanner.

- 4 For Contact Mode, the dotted yellow line (Figure 65) shows the recommended vertical alignment of the laser prior to approaching the sample.

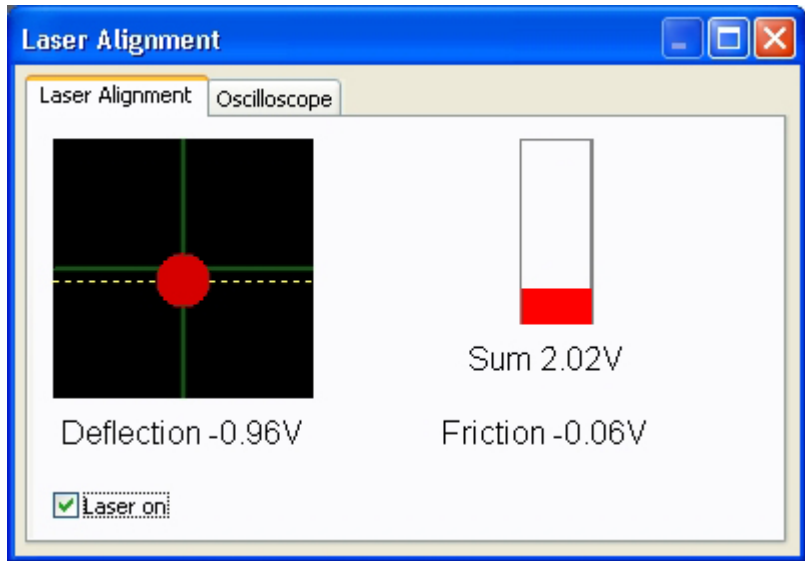


Figure 65 Align spot to yellow, dotted line for Contact Mode

Mounting the Sample

The Keysight 5500 SPM accepts a wide variety of sample plates, including specialized plates for imaging in liquid, in controlled temperature, etc. To use a sample plate:

- 1 Mount the sample to the sample plate. In general, samples should be held in place securely enough to prevent drift or creep during measurement, but not so firmly as to induce stress in the sample. Several mounting methods are available. A common approach is to mount the sample on a magnetically attractive backing which can then be held by the magnet on the standard sample plate. Large, flat samples can be held down using the clips from the liquid cell plate. Double-back tape can also be used, though the tape tends to deform easily and can lead to creep during imaging.

CAUTION

Verify that there is enough space below the scanner and probe that they will not contact the sample plate once it is mounted. Contact with the plate will damage the probe and sample and could also damage the scanner.

It is recommended that you move the scanner up 100 microns or more whenever changing a probe or plate to avoid damage.

- 2 Place the sample plate's front alignment tab over the front alignment pin, as in image A of [Figure 66](#).

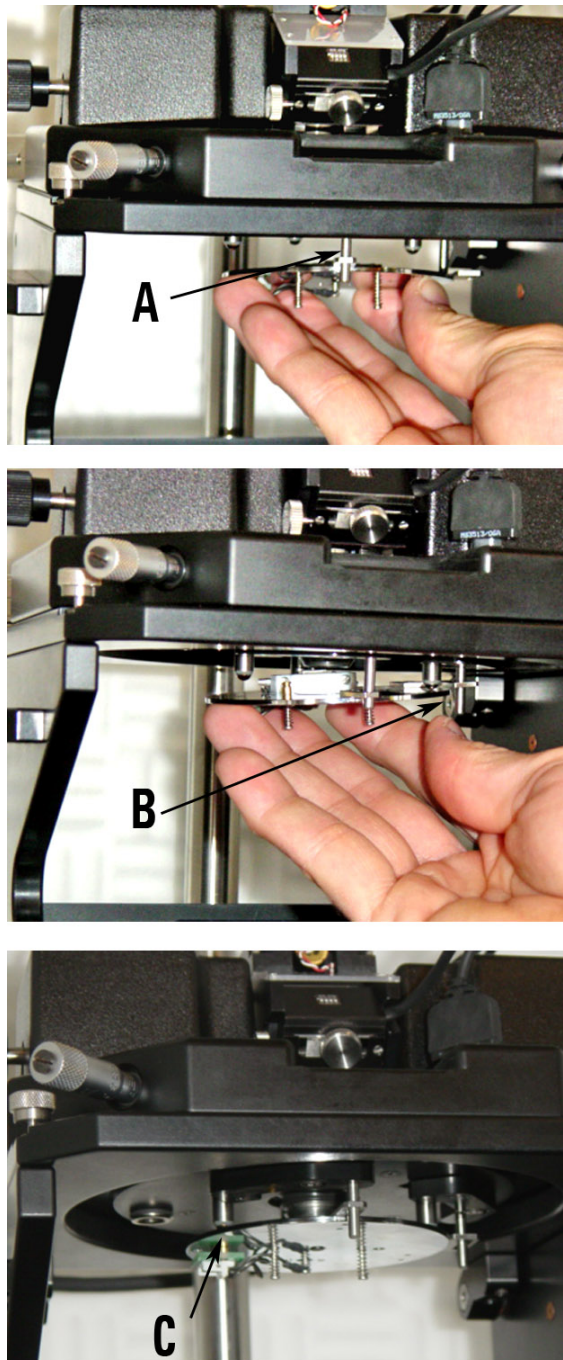


Figure 66 Mounting a sample plate.

- 3 Place the second alignment tab over the alignment pin as in image B of [Figure 66](#).

- 4 Let the magnets on the three posts gently engage the sample plate to hold it in place, as in image C of [Figure 66](#).

CAUTION

The sample plate magnets are quite strong and, if allowed to, will snap the sample plate into position, which may perturb the sample. Holding the plate at the edges while engaging the magnets will control this movement.

- 5 For certain measurements, you will need to connect the EC/MAC cable ([Figure 67](#)) from the sample plate to the socket on the underside of the microscope stand. The 3-pin EC cable provides bias to the sample. The 3-pin MAC connector provides signal to the magnetic coil for MAC Mode.

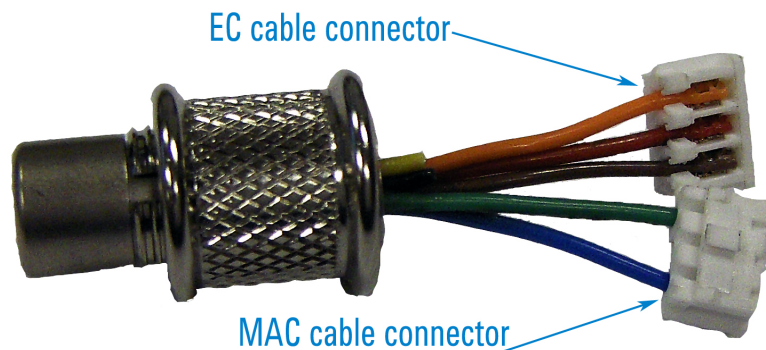


Figure 67 EC/MAC cable

Using the Video System

The Keysight 5500 SPM includes a USB-based video system for viewing the tip and sample. The video system's optics and optics in the scanner together provide optical magnification of 3.8X - 24.3X to the camera.

The video system's illuminator is a separate box (Figure 68) connected to the video system by a fiber light pipe. The light pipe can be separated from the illuminator and/or optics by loosening the knobs at either of its ends. Turn on the illuminator with the switch on the front of the illuminator box. Increase intensity by turning the Illumination Level knob clockwise, or decrease it by turning it counterclockwise.

NOTE

The video system camera automatically adjusts to the contrast level. Above a certain level turning up the illumination will no longer have any effect, as the camera will compensate to counteract the increased intensity.



Figure 68 Turn on the illuminator and adjust intensity

NOTE

For critical measurements use the video system to align the sample, then turn off the illuminator to minimize system noise.

The z-position knob (Figure 69) lets you raise and lower the video system optics. Typically the optics are situated 3.5 inches above the scanner; this position is set by a stop ring on the pole. You may need to adjust this level to accommodate the Environmental Chamber or other optional hardware.



Figure 69 Adjusting the z-position

To adjust the lateral position of the video system relative to the scanner, turn the knobs on the front and right side of the optics assembly (Figure 70). Turn the front knob clockwise to move the field-of-view backward, and counterclockwise to move it forward. Turn the side knob

clockwise to move the field-of-view to the right, and counterclockwise to move left.

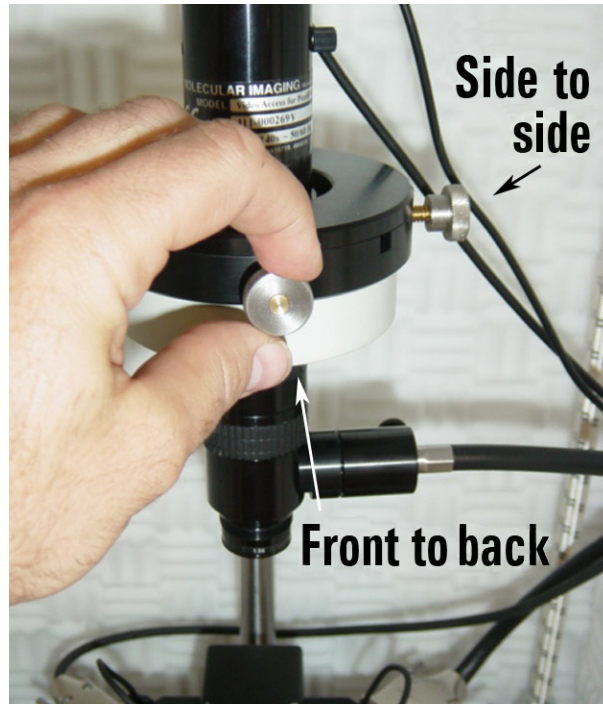


Figure 70 Adjusting lateral position of the video system

Twist the Zoom control ([Figure 71](#)) to the left to increase the zoom, or to the right to decrease zoom.



Figure 71 Adjusting zoom

Choose **Controls > CameraView** to view the video output from the camera (Figure 72).

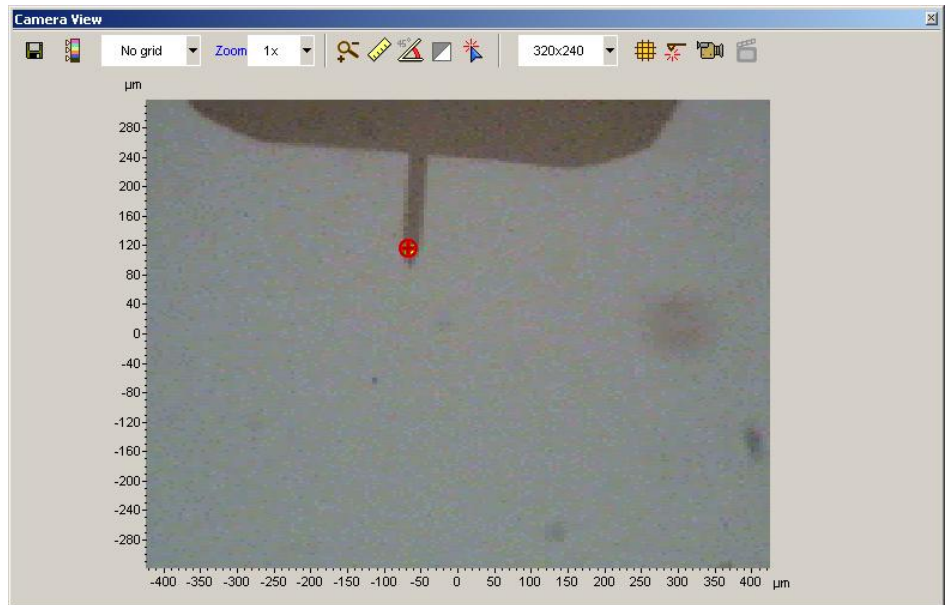


Figure 72 Camera View window showing tip and sample.

5 Contact Mode Imaging

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In Contact Mode imaging, the AFM tip is brought into gentle contact with the sample and then scanned in raster fashion across the sample surface. The system will either maintain a constant force on the tip, for most Contact Mode measurements, or will maintain the tip at a constant height, for high resolution imaging of very flat surfaces. It is typical in Contact Mode to image deflection, friction and/or topography, though other signals may be imaged as well.

In this chapter we will outline the steps to making Contact Mode images with a system that is calibrated and properly set up. Additional factors may affect the quality of images produced in Contact Mode. To understand more about these factors please be sure to read the PicoView software documentation and Keysight training materials.

NOTE

This chapter references material in [Chapter 4](#), "Preparing for Imaging." Be sure to review and understand [Chapter 4](#) before continuing with Contact Mode.

Setting Up for Contact Mode Imaging

Contact Mode imaging can be completed with any of the multi-purpose scanners, using most any AFM probe and nose assembly. Contact Mode tips, however, are designed specifically for this application, with lower resonance frequency, softer cantilevers.

Constant Force Mode

In Constant Force Mode, a feedback loop between the Head Electronics Box (HEB) and the controller maintains a constant deflection of the tip based on the specified Setpoint voltage. The error signal, which is the difference, measured in volts by the photodetector, between the Setpoint and actual cantilever deflection, is read as the Deflection.

To begin imaging, follow the steps you learned in [Chapter 4](#):

- 1 Insert the nose assembly into the scanner.
- 2 Insert a probe into the nose assembly.
- 3 Place the scanner in the microscope and connect its cables.
- 4 Align the laser on the cantilever.
- 5 Insert and align the detector.
- 6 Prepare the sample and mount the sample plate.

Then:

- 7 In the PicoView software choose **Mode > Contact**.
- 8 Choose **Controls > Camera View** to open the Camera View video window.
- 9 Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.
- 10 Viewing the video window, bring the tip and sample very close to contact:
 - c Adjust the focus and x-y alignment of the video system such that the tip is in sharp focus ([Figure 73](#)).

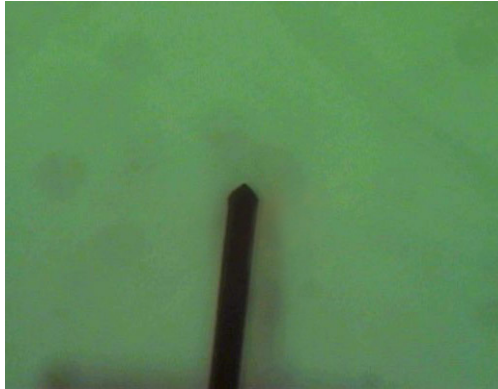


Figure 73 Tip in focus through video system

- d Lower the focal plane just slightly below the tip by turning the Z-position control toward you until the tip is slightly out of focus ([Figure 74](#)).

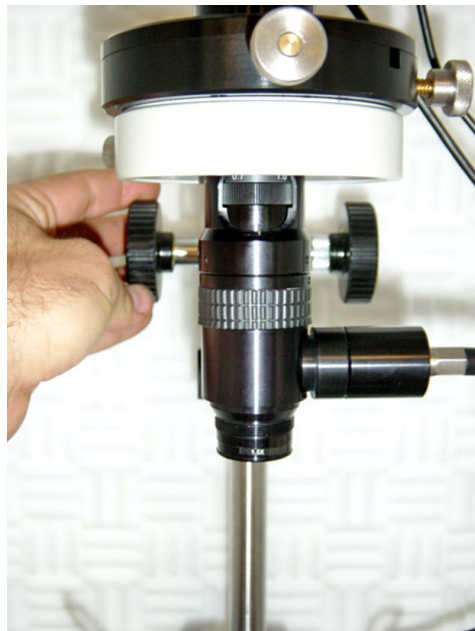


Figure 74 Lower focal plane just below tip

- e Using the **Close** switch on the HEB, raise the sample until the sample comes nearly into focus. The tip should now be just above the sample surface.

CAUTION

Raise the sample slowly and carefully to avoid collision with the sample. Hard contact between the tip and the sample can damage either or both.

- 11 Locate the area of interest on the sample by manually moving the X/Y stage control micrometers (Figure 75) while watching the video window.

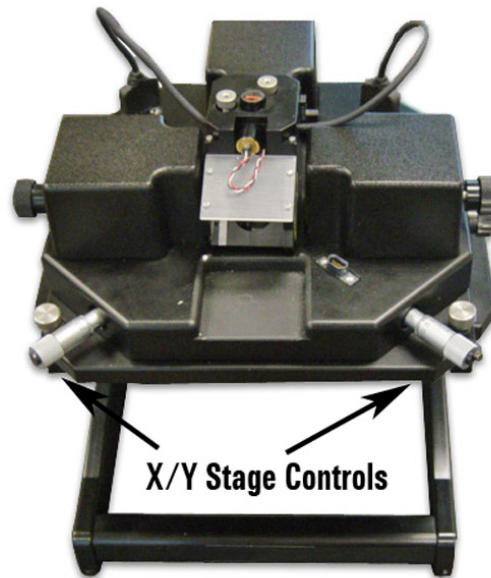


Figure 75 Stage control micrometers

CAUTION

If your sample has tall features or steps, you may need to raise the scanner slightly to avoid contacting features as you move the stage.

- 12 Next you will “approach” the sample, bringing the tip into contact with the surface. To ensure that the contact will be gentle, verify that the Setpoint voltage is set appropriately:
 - a Note the **Deflection** reading on the HEB front panel, or in PicoView’s Laser Alignment window (both will display the same value). This is the current cantilever deflection, stated in volts.
 - b In PicoView’s Servo window, enter a **Setpoint** value slightly more positive than the current Deflection reading. This sets the deflection that the feedback loop will achieve and maintain.

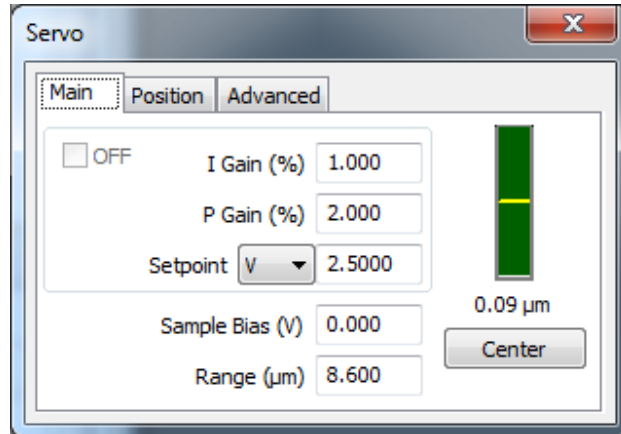



Figure 76 Servo window showing Setpoint voltage and Gains

NOTE

If the Servo window is not already open, choose **Controls > Imaging** to open it. The Scan and Motor and Realtime Images windows will also open at the same time.

- 13 Click the **Approach** button in PicoView's toolbar . The system will raise the sample until the deflection reaches the Setpoint value.

NOTE

The **Approach Range**, the distance that the system will move the scanner to try to contact the surface, is set in the Microscope Setup window (**Controls > Setup > Microscope**).

The indicator on the right side of the servo window shows the possible displacement range for the Z piezo actuator. The indicator will be red when the scanner is too far from (or too close to) the surface for the system to maintain the Setpoint. The indicator will turn green when contact is made and the Setpoint is maintained. A yellow bar will show the position of the piezo within its available range of motion.

The center of the range is defined as “zero,” with positive values indicating piezo displacement away from the sample, and negative values being toward the sample. In **Figure 76**, the positive voltage shows that the piezo is maintaining the setpoint while it is slightly above the center point of its range.

- 14 Also in the Servo window make sure that the I Gain and P Gain are set to **10 %**. These gains dictate how quickly the system will react to changes in tip deflection in order to maintain constant force. 10 % is

a good starting value; more information on optimizing the gains is in “Gains” on page 114.

- 15 In the Scan and Motor window, select the **Scan** tab (Figure 77).

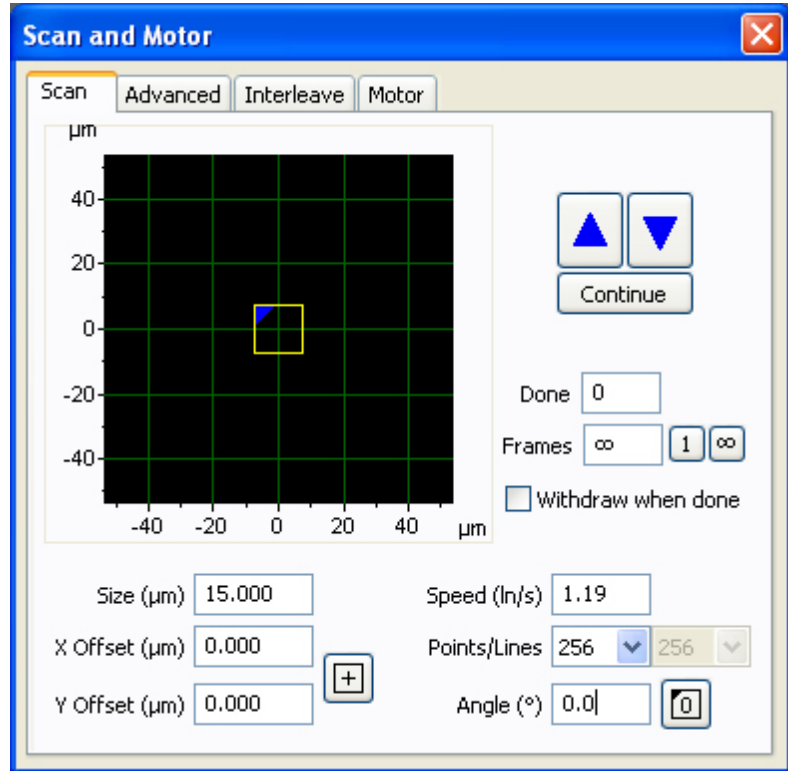


Figure 77 Scan and Motor window: Scan tab

- 16 Enter a scan **Speed**, stated in Lines/Second (ln/s). A typical starting value is 1-2 ln/s.
- 17 Select a resolution from the **X** list. **256** is a good starting value, providing ~11 nm/pixel resolution for the 3 micron scan size selected in Figure 77.
- 18 The grid in the Scan and Motor window shows the range of motion of the X and Y piezo actuators. The yellow square represents the size and location of the scan, based on the current scan settings. Change the **Size** (in microns) to set the scan size in both X and Y. Enter **X Offset** and/or **Y Offset** values to move the scan region. You can also click and drag the yellow box to move the region. Click the “+” button to return the offsets to zero.
- 19 In the Realtime Images window, choose to display Topography, Deflection and Friction. Click the “+” button of the window to add a buffer. To set what each buffer will display, **right-click** inside the

buffer frame, then select the **Input Signal** from the list (Figure 78). The list will vary depending on the imaging mode.

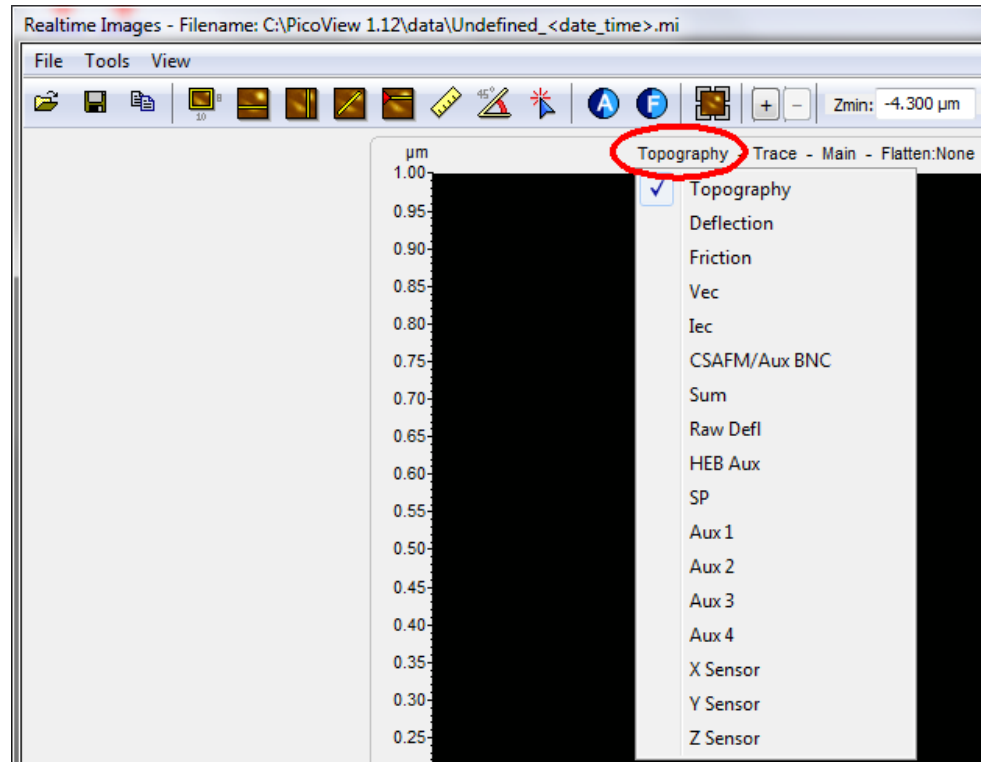


Figure 78 Selecting the Input signals in the Realtime Images window

20 In the Scan and Motor window, click the **Down** blue arrow to initiate a scan starting at the top of the grid. Click the **Up** blue arrow to initiate the scan from the bottom up (Figure 79). The image maps will begin to be rendered in the Realtime Images window.

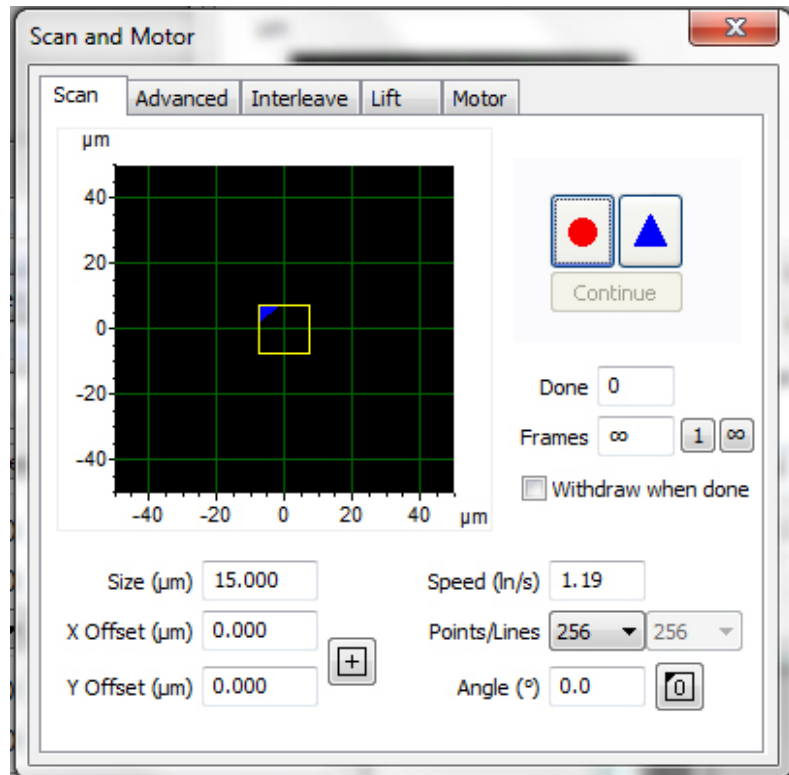


Figure 79 Scan and Motor window after scan has been initiated

As the tip moves across the first scan line, the system will adjust the voltage on the z-piezo actuator to maintain constant force (as specified by the Setpoint value).

NOTE

The important parameter is the difference between the Deflection setting (shown on the HEB) before beginning the approach and the initial Setpoint value. A Setpoint of +1 V could be too low if the initial Deflection was -0.1 V but too high if the initial Deflection reading was -2 V.

For each pixel, the system will record and plot the error signal (the difference in volts between the surface-induced deflection and the Setpoint) as the Deflection Image (in volts).

The correction signal (the voltage that the feedback loop applies to the z-piezo to maintain the deflection at the Setpoint) is scaled by the piezo sensitivity (nm/V) and plotted as Topography (in nanometers).

As the tip passes over regions of varying friction it will twist in the scan direction as well as deflecting in the vertical axis. The detector senses

change in the cantilever's twisting motion and outputs it as the lateral deflection (Friction) signal, which is plotted as the Friction image (in volts). Changes in lateral force on the tip can be caused either by changes in frictional properties across the sample or by variations in topography. The Friction signal will therefore be a convolution of these two components. Comparing the friction and topography images helps to differentiate the impact of topography versus friction.

At the end of each scan line the system will "retrace" the scan line until it once again reaches the beginning. The scanner will then advance one line width and another line will be scanned.

Depending on the Frames setting in the Scan and Motor window, the system will either scan once and stop, or it will scan infinitely, overwriting the previous scan each time. You can also choose to scan for a specific number of frames. To stop the scan, click the red STOP circle that will replace the Up or Down arrow when you start a scan (Figure 77 on page 110).

Constant Height Mode

In Constant Height Mode the system maintains the tip in a plane above the surface. It is functionally the same as Constant Force Mode, except that the feedback circuit gains are set very low so that the system does not react to changes in tip deflection.

To image in Constant Height Mode, in the Servo window set the I and P gains to 0.1 %. This will effectively cause the system to no longer adjust the tip force. This lack of feedback reduces signal noise, enabling atomic-level resolution imaging of very flat samples. The scan speed can also be faster since the system will no longer attempt to react to changes in deflection.

The error signal (in volts) is used to generate an image that is sensitive to small changes in topography.

Fine-Tuning the Image

Besides the sharpness of the scanning tip, the quality of imaging in Contact Mode is largely dependent on three factors: the Setpoint voltage, feedback gains, and scan settings.

Setpoint

When the Setpoint is too negative, the system will continue as if contact is established between the tip and sample even if it actually is not. In

this case, the tip will not accurately trace surface topography—in the extreme, the topography image will appear entirely flat. Making the Setpoint more positive increases the force applied to the sample by the tip. Higher force can place undue wear on the tip and, in the extreme, can damage the tip or sample.

The optimal Setpoint value, which will vary per sample and per probe, places enough force on the tip to accurately trace the topography without placing unnecessary force on the tip. A good method for setting the Setpoint is as follows:

- 1 With your cursor still in the Setpoint box, press the **Down** arrow on your keyboard to make the Setpoint more negative. At some point, the Setpoint voltage will drop so low that the tip will leave contact with the sample—when it breaks free, the indicator in the Servo window will change from green to red.
- 2 Pressing the **Up** arrow on your keyboard, raise the Setpoint again until the tip just regains contact with the sample. This is the lowest possible force that will keep the tip and sample in contact.
- 3 During the scan, you may choose to raise the Setpoint to improve tracking of the topography. Some iteration may be required to reach the optimal value.

Gains

The I and P gains in the Servo window dictate how quickly the feedback system reacts to changes in deflection. Typically the I (Integral) and P (Proportional) gains are set to the same value; the I gain setting has a much greater effect on imaging than the P gain.

When the gains are set too high, the system will overcompensate to correct changes in tip deflection which will lead to “ringing” at the leading and trailing edges of features. When the gains are too low, the system will not adjust the tip quickly enough for that scan speed, blurring the topography.

The following procedure is a useful way to adjust the gains:

- 1 Begin imaging with **I Gain** and **P Gain** set fairly low (0.50-1.00).
- 2 Slowly increase the **I Gain** to increase the contrast and detail of the image.
- 3 Increase the **I Gain** until oscillations appear in the scan lines, then decrease until the oscillations cease.
- 4 Adjust the **P Gain** to increase image quality. P Gain is typically larger than I Gain, almost 2X or more depending on conditions.

Gain settings of 5-10 % are typical, though some iteration will most often be required to optimize the gains for a particular sample.

Scan Settings

In the Scan and Motor window, the scan Speed and Resolution (X) will all affect image quality.

A faster scan speed decreases imaging time but may not allow the system sufficient time to accurately track the topography. A typical scan speed will be 2-5 lines/second for smooth surfaces. For rougher surfaces a lower scan speed may be needed.

A typical resolution of 256 pixels/line provides good resolution and speed. Increasing the resolution will improve image quality but will require longer imaging times.

One good option is to scan a large region at low resolution and high speed, and then to zoom in on a region of interest for a high resolution scan at lower speed. After completing the large scan, use the Offset and Size values in the Scan and Motor window to adjust the scan to cover the region of interest. Increase the Resolution, decrease the Speed, and re-scan the zoomed region.

6 AC Modes

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In AC Mode, introduced in “[Intermittent Contact AFM](#)” on page 23, a sinusoidal voltage is applied to a piezo element or magnetic coil in the nose assembly or sample plate. The piezo or magnetic coil causes the probe tip to oscillate, typically at or near one of its resonance frequencies, such that it taps gently on the surface. The tip is then raster-scanned over the region of interest while the amplitude of oscillation is monitored to produce images. Through this method, lateral forces on the tip are virtually eliminated, enabling higher resolution imaging than is possible with Contact Mode.

NOTE

This chapter references material in [Chapter 4](#) and [Chapter 5](#). Be sure to review and understand [Chapter 5](#) before continuing with AC Mode.

The process for imaging in AC Mode is similar to that of Contact Mode, with one additional step: the cantilever must be tuned to near its resonance frequency.

There are two methods for providing the oscillation: Acoustic (AAC) and Magnetic (MAC). Both AAC and MAC Modes require the additional MAC Mode or MAC III controller.

The MAC controllers utilize “lock-in amplifier” technology to precisely determine the oscillation amplitude and phase response of the cantilever, resulting in excellent force regulation and high-quality phase

images. The phase conversion coefficient is 18° per volt for the MAC Mode and MAC III controllers.

To use a MAC controller, choose **Controls > Setup > Components** and verify that the **Serial Port AC Mode Controller** check box is selected.

Acoustic AC Mode (AAC)

In Acoustic AC (AAC) Mode AFM, a piezo-electric transducer in the nose assembly drives the cantilever oscillation. Note that the nose assembly (Figure 80) includes two contact pins through which the drive signal is routed to the transducer. AAC Mode probe cantilevers have resonance frequencies typically in the 100-300 kHz range. Any sample plate can be used.



Figure 80 AAC Mode nose assembly

AAC Mode

- 1 To image in AAC Mode, first follow the steps from [Chapter 4](#):
 - a Insert the nose assembly into the scanner.
 - b Insert a probe into the nose assembly.
 - c Place the scanner in the microscope base.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
 - f Prepare the sample and place it on the sample plate.
 - g Adjust the video system to focus on the cantilever.
- 2 Use the manual screws for coarse approach.
- 3 Use the **Close** switch on the HEB for a final approach to bring the tip close to, but not touching, the sample.
- 4 In PicoView choose **Mode > ACAFM**.

- 5 Choose **Controls > AC Mode** to open the ACAFM Controls window (Figure 81).

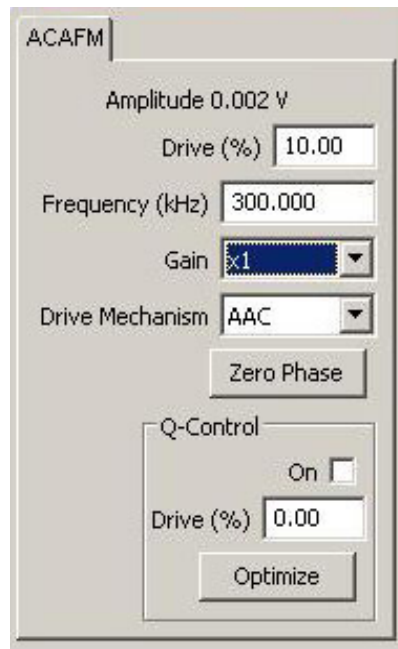


Figure 81 ACAFM Controls window

- 6 Set the Drive Mechanism to **AAC**.
- 7 Set the Drive (%) to **10 %**. This is the amplitude of the AC drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.

NOTE

If the system is using the AC source in the HEB, the Setpoint needs to be set to zero to perform Auto Tune. If the system has an external AC/MAC box, the Setpoint can be any value during Auto Tune.

- 8 Choose **Controls > AC Mode Tune** to open the AC Tune window (Figure 82).

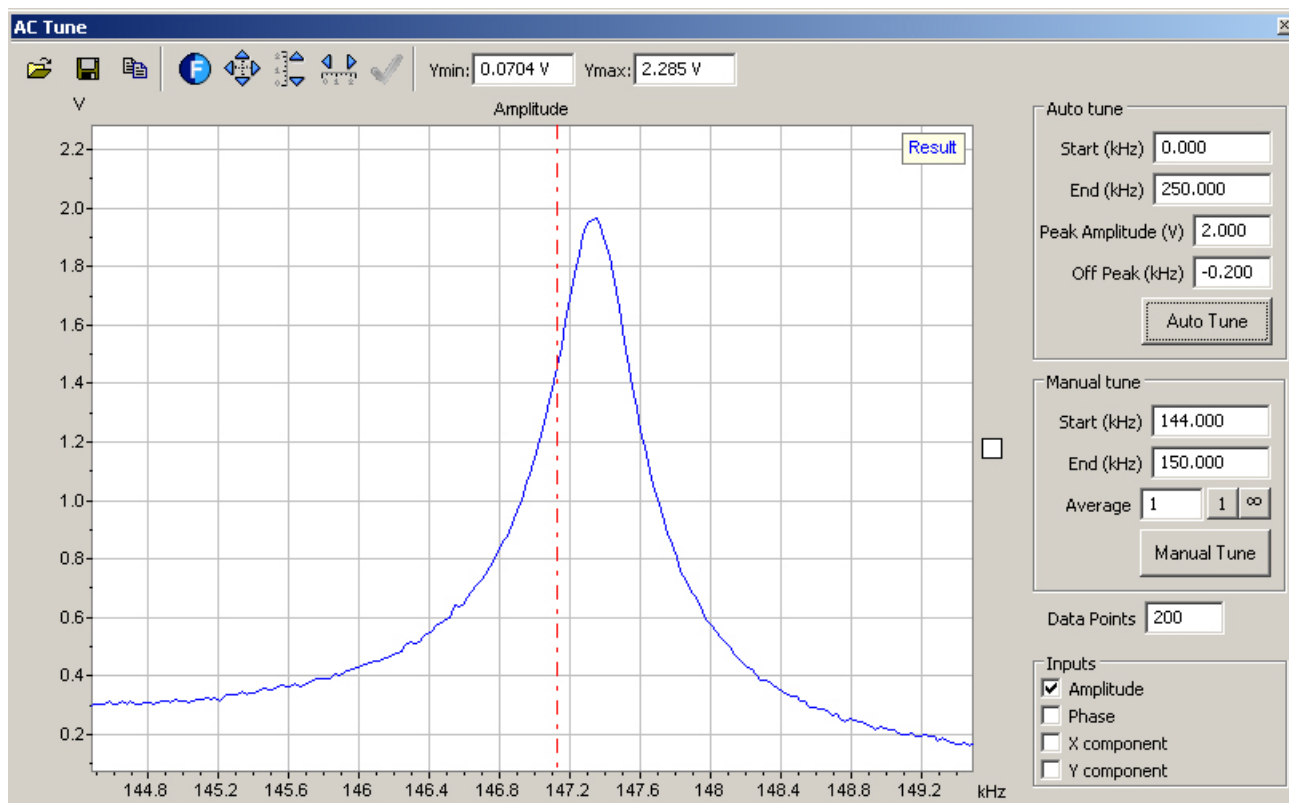


Figure 82 AC Tune window with resonance peak at ~147 kHz

The next step is to tune the oscillation signal to match the frequency of the particular cantilever. You will use the controls in the AC Tune window to sweep through a range of frequencies. The resultant plot should show one strong, sharp resonance peak. The cantilever's storage box should indicate the range in which the primary resonance frequency will be found.

- 9 In the upper-right Auto Tune area of the AC Tune window, enter the **Start** and **End** frequencies (in kHz) for the tuning sweep. For a new or unknown cantilever, use the stated minimum and maximum frequencies given on the storage box. If you happen to know the resonance frequency more exactly, you can use a smaller range to speed the tuning process.
- 10 Set the Peak Amplitude, the maximum desired amplitude of cantilever oscillation. 2 volts is a typical starting value.
- 11 To ensure good engagement of the tip with the sample, set the oscillation Frequency slightly below the actual resonance frequency

of the cantilever. Enter an Off Peak value to offset the oscillation frequency from the cantilever's resonance frequency. A typical starting value is **-0.200 kHz**.

- 12 Click the **Auto Tune** button. The system will sweep several times through the range of frequencies, locating the peak oscillation amplitude within the range (Figure 82). The AC signal oscillation will be set to this value, taking into account the specified Offset.
- 13 Focus the cantilever in the video window.
- 14 Turn the video system focus knob toward you such that the tip goes just out of focus (the focal plane is just below the tip now).
- 15 Press the **Close** switch to raise the sample until both the tip and sample are in focus (i.e., they are nearly touching).

Next, bring the tip into contact with the sample. In AAC mode, "contact" occurs when the cantilever oscillation is dampened to a specified percentage of the total oscillation.

- 16 In the Scan and Motor window, click the **Motor** tab (Figure 83).

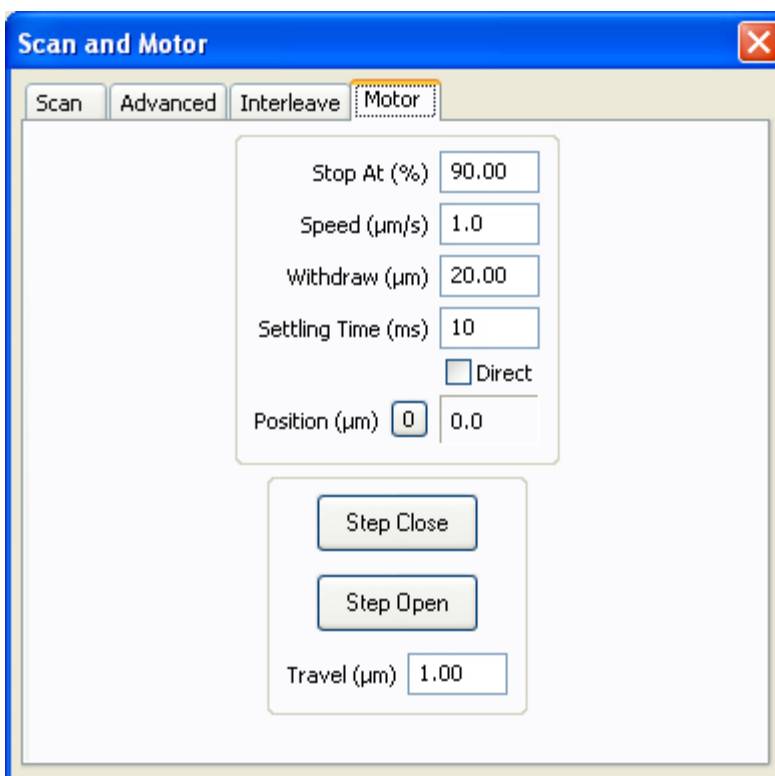



Figure 83 Set the Stop At value in the Scan and Motor window

- 17 Set the Stop At (%) to specify the percentage of total oscillation that represents "contact." For example, if the total oscillation amplitude

(set in [step 9](#)) is 2 volts, and the Stop At value is set to **90 %**, the approach will stop when the oscillation is damped to 1.8 volts.



- 18 Click the **Approach** button in PicoView's toolbar . The system will raise the sample until the amplitude is damped to the Stop At percentage. Because of air damping, oscillation typically decreases as the tip nears the sample. The software monitors the rate of change of amplitude as well as the absolute value, so the final amplitude will not be exactly the Stop At percentage.
- 19 In the Servo window set the I Gain and P Gain to **5 %**. These gains dictate how quickly the system will react to changes in amplitude.
- 20 In the Scan and Motor window, select the **Scan** tab.
- 21 Enter a scan Speed of **1-2** In/s and a Resolution of **256**. More information on fine tuning these settings can be found in [Chapter 5](#).
- 22 Enter the **Size** (in microns) and **X Offset** and/or **Y Offset** values to set the size and center of the scan. You can also click and drag the Scan box in the graph on the Scan and Motor window to adjust the scan size and location.
- 23 In the Realtime Images window, make sure that **Topography** and **Deflection** are displayed.
- 24 In the Scan and Motor window, click the **Down** blue arrow to initiate a scan starting at the top of the grid. Click the **Up** blue arrow to initiate the scan from the bottom up. The image maps will begin to be rendered in the Realtime Images window.

As the tip moves across the first scan line, the system will adjust the voltage on the z-piezo to maintain constant amplitude (as specified by the Setpoint voltage).

For each pixel, the system will record and plot the error signal (the amount the oscillation amplitude would deviate from the Setpoint voltage as the Amplitude Image (in volts).

The correction signal (the voltage that the system applies to the z piezo to maintain the amplitude) is scaled by the system sensitivity and plotted as topography (in nanometers).

At the end of each scan line the system will “retrace” the line to the beginning of the scan. The scanner will advance one line width (based on the resolution setting) and another line will be scanned.

Depending on the Frames setting in the Scan and Motor window, the system will either scan once and stop, or it will scan infinitely,

overwriting the last scan each time. You can also specify a specific number of scans to complete.

To stop the scan cycle, click the red **STOP** circle that will replace the Up or Down arrow when you start a scan.

Constant Height Mode

AC Mode imaging can be completed using either Constant Amplitude mode, as described above, or Constant Height mode. In Constant Height mode the tip remains in the same horizontal plane throughout the scan (it does oscillate, but the center of that oscillation stays in plane). The servo gains are set very low so that the system effectively does not react to changes in amplitude.

Constant Height Mode should only be used for very flat samples. To image in Constant Height mode, in the Servo window set the I and P gains to 0.1 %. This system will only very slowly adjust the z-piezo in response to amplitude changes. This lack of feedback reduces signal noise, enabling high resolution imaging of very flat samples.

The change in amplitude as the tip scans across the sample is mapped as Amplitude and displayed in volts in the Image buffer.

Magnetic AC (MAC) Mode

In Magnetic AC (MAC) Mode AFM, a cantilever coated in magnetic material is driven by a coil-generated oscillating magnetic field. The Lock-in in the MAC controller precisely determines and maintains oscillation amplitude and phase relation changes.

In AAC Mode, the oscillator in the nose assembly oscillates the entire system, including liquid if imaging in liquid. In MAC mode, because the oscillation is driven by a magnetic field, only the magnetically-coated tip oscillates, providing a sharper resonance peak, and therefore higher resolution imaging. MAC Mode is the most accurate AC technique available, particularly for imaging in liquids.

MAC Mode requires either a MAC or MAC III controller, both of which offer the lock-in amplifier required to precisely drive the MAC coil (the

MAC III offers additional lock-in amplifiers for other more complex modes as well). Specially coated MAC cantilevers are also required.

Standard MAC Mode

In standard MAC Mode, the coil is located in the sample plate (Figure 84). A Contact Mode or AC Mode nose assembly can be used.

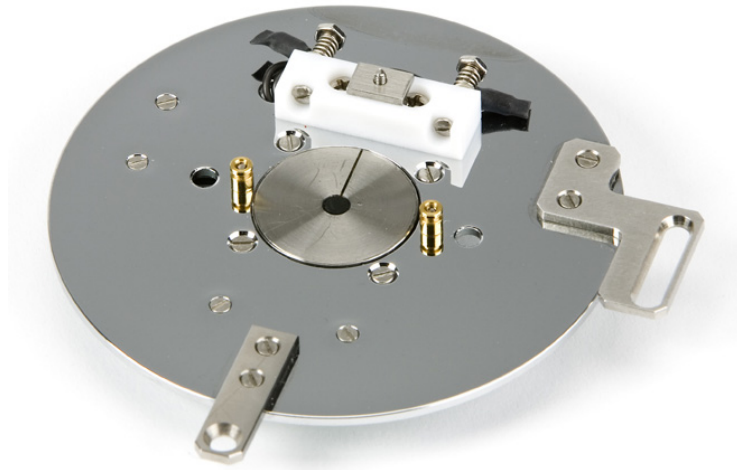


Figure 84 MAC Mode sample plate

The procedure for imaging in MAC Mode is the same as for AC Mode, with these exceptions:

- 1 Plug the **EC/MAC cable** socket to the bottom of the microscope stand.

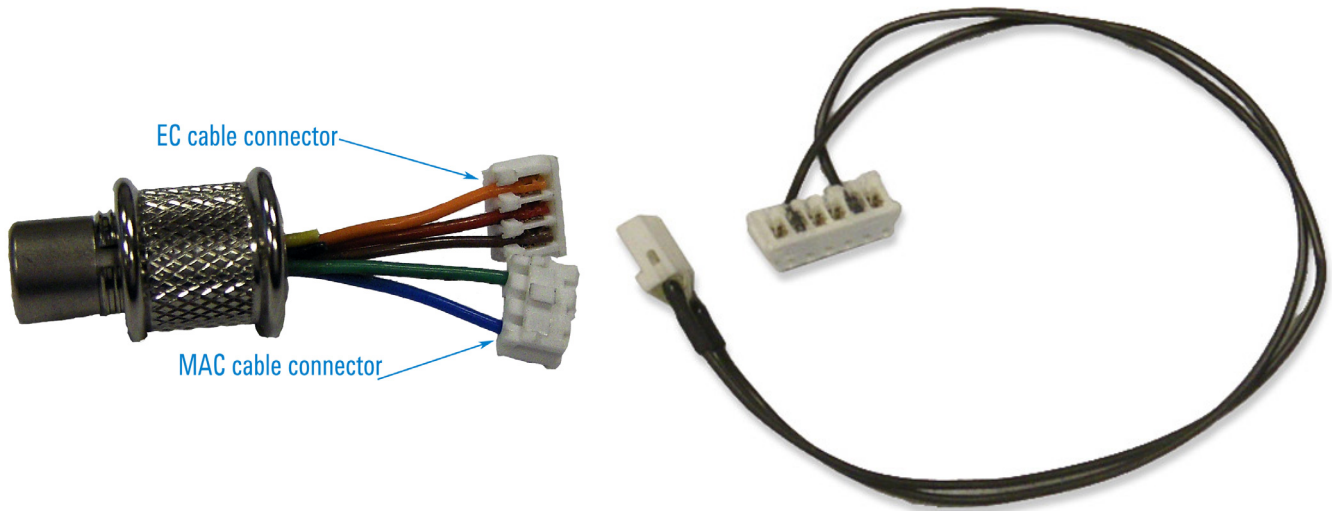


Figure 85 EC/MAC cable (left) and MAC cable (right)

- 2 Connect the MAC cable connector (blue and green wires) of the EC/MAC cable to the 3-pin connector on the MAC cable (Figure 85). Connect the MAC cable to the 6-pin connector on the sample plate as shown in Figure 86 on page 126.

- 3 If a sample bias is needed, connect the EC cable connector to the EC cable connected to the 3-pin connector on the MAC sample plate, as shown in [Figure 86](#).

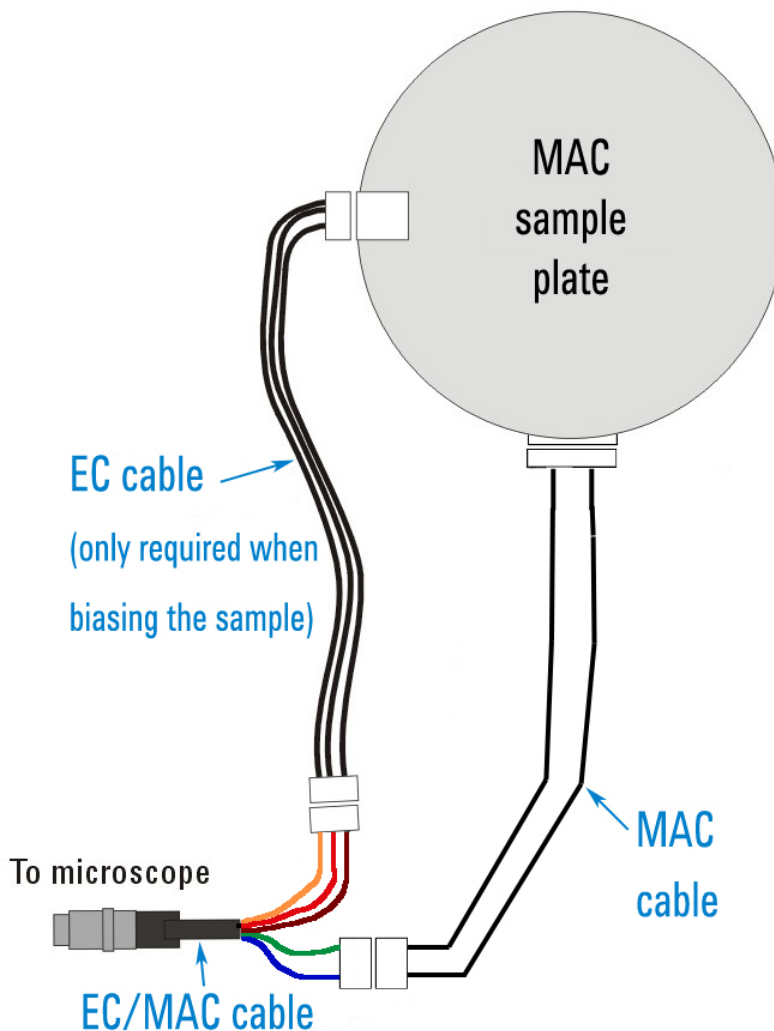


Figure 86 Cable connections for MAC mode

- 4 In the ACAFM Controls dialog box, choose **MAC** as the Drive Mechanism.

Top MAC Mode

In Top MAC Mode AFM, the driver coil is located in the nose assembly ([Figure 87](#)). This configuration provides better tip response when

imaging thick samples which can lessen the magnetic field oscillating the tip. Any sample plate can be used for Top MAC imaging.



Figure 87 Top MAC nose assembly

Q Control

An oscillating cantilever in AC Mode is influenced by complex interaction forces between it and the surface. By carefully setting system parameters the system can be made to operate entirely in the regime of net-attractive forces, thereby reducing the effect of the probe tip on the sample. The range in which the parameters have to be adjusted can be narrow, however, making it difficult to maintain in real operation.

Q Control is a method that reduces damping of the system, increasing the quality factor of the oscillating cantilever. Enhanced resonance allows imaging with very low force and high phase sensitivity. The

well-defined resonant peak in MAC Mode makes the method particularly effective.

Q Control uses a feedback loop to alter the sharpness of the resonance peak. It is only available with the MAC III controller, and it can be used with either AC Mode or MAC Mode.

To use Q Control:

- 1 Adjust the Drive % to **0.2 %** for Lock-in 1 in the upper section of the Main tab the ACAFM Controls window (Figure 88, on left).
- 2 Run a standard AAC Mode **Auto Tune**. This will update the Amplitude, Drive % and Frequency as shown in the ACAFM Controls window on the right in Figure 88.

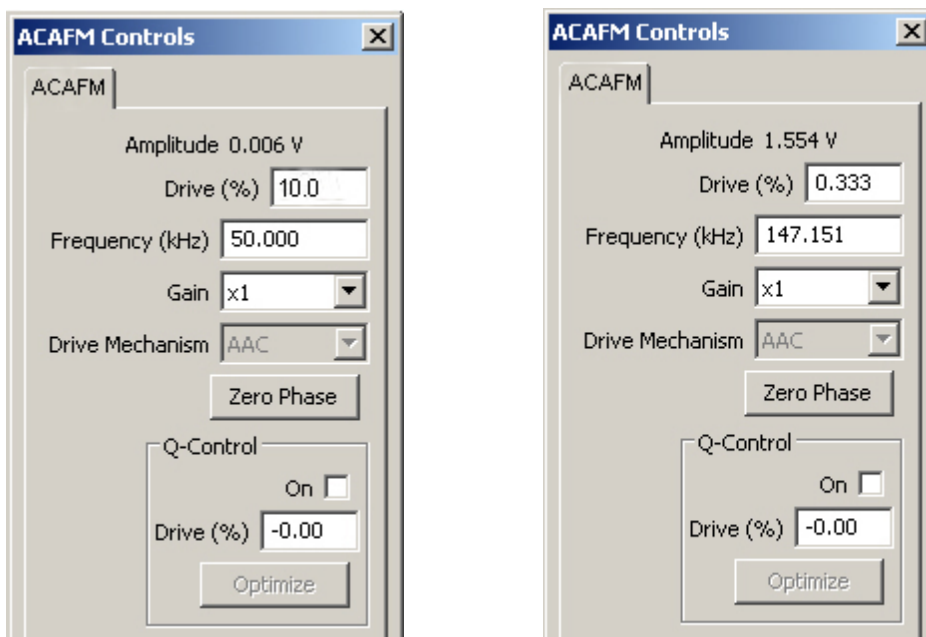


Figure 88 Q Control settings in the ACAFM Controls window before (left) and after (right) Auto Tune

- 3 Select the Q Control **On** check box.
- 4 Click the **Optimize** button.

This will set the optimal Q Control Drive (%) value in the lower section of the ACAFM Controls window as shown on the left in Figure 89 on page 129. The optimized Drive (%) and Phase Shift will also show in

the Other tab of the Advance AC Mode Controls window, shown below in [Figure 89](#) with the Q Control parameters circled in red.

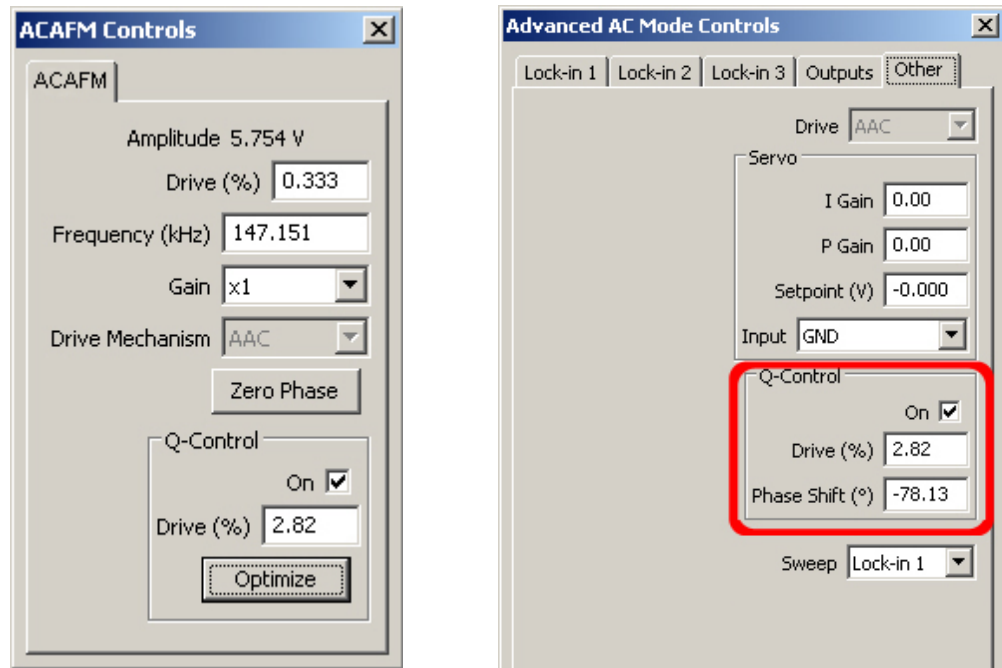


Figure 89 ACAFM Controls and Other tab after Q control optimization

Note that negative numbers can be used for the Q Control Drive (%) to lower the Q Control drive amplitude. However the standard AC Mode Drive (%) must be a positive number.

Click **Manual Tune** over the same frequency range as in **step 2** to generate a new plot using the values from the Q Control optimization.

Figure 90 below shows the change in the amplitude resonance peak before (top) and after (bottom) applying Q Control.

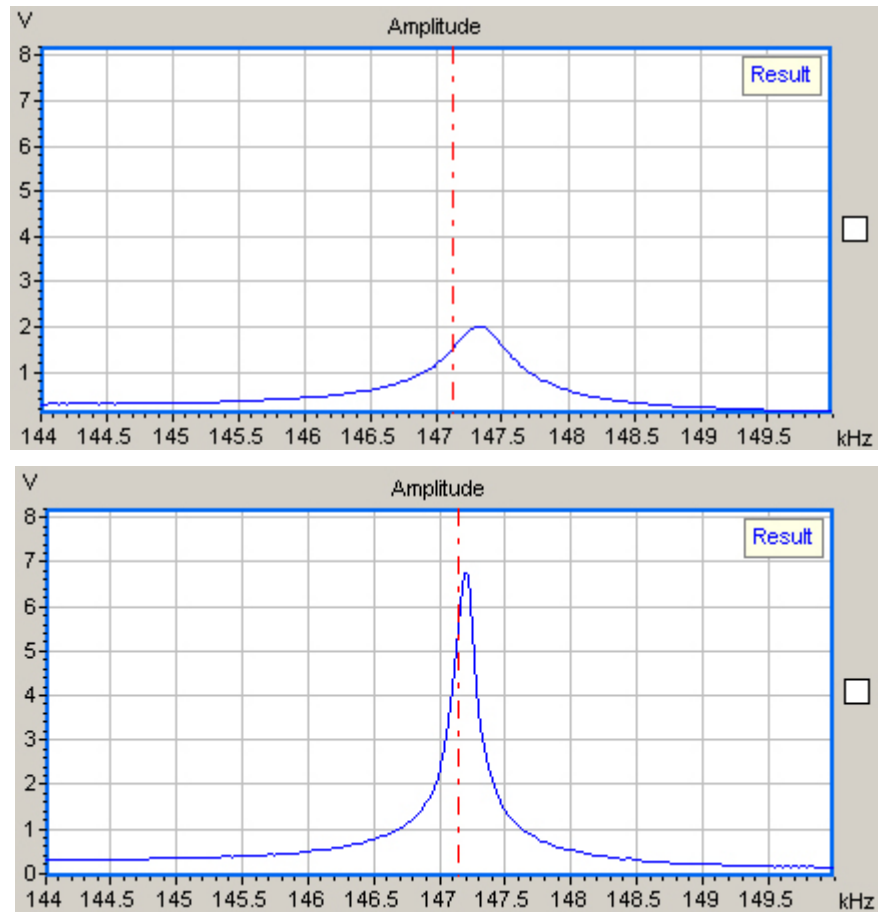


Figure 90 Amplitude resonance peak before (top) and after (bottom) optimizing Q Control

Note the phase curve will also show a steeper slope as shown below in Figure 91.

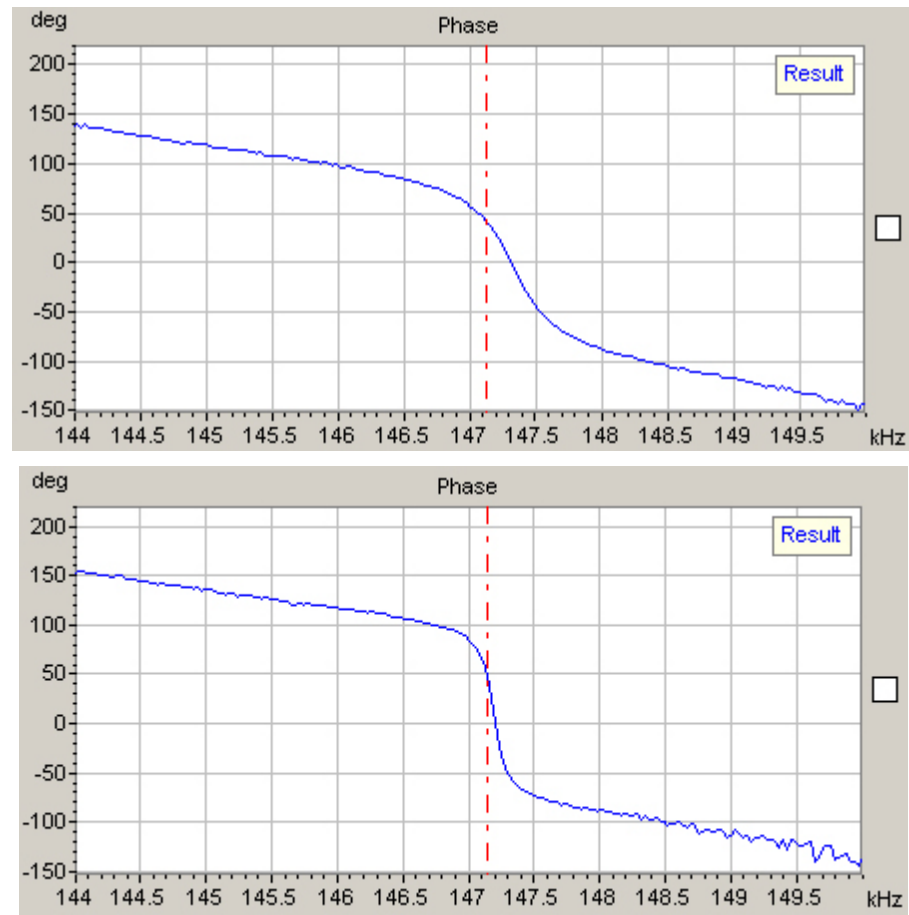


Figure 91 Phase before (top) and after (bottom) optimizing Q Control

7 Additional Imaging Modes

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One of the primary advantages of the Keysight 5500 SPM is that it allows you to perform many different imaging modes with the same basic hardware. Most of the modes presented in this chapter are based on Contact Mode or AC Mode imaging, so be sure that you have read the information in [Chapter 4](#), [Chapter 5](#), and [Chapter 6](#) before proceeding with this chapter.

CAUTION

Some nose assemblies, such as the two-piece nose assemblies and CSAFM nose assembly, are not sealed and should never be used for imaging in liquid. Be sure to use only approved nose assemblies for imaging with the liquid cell.

Scanning Tunneling Microscopy (STM)

In STM, a bias voltage is applied between a sharp, conducting tip and the sample. When the tip approaches the sample, electrons “tunnel” through the narrow gap, either from the sample to the tip or vice versa, depending on the bias voltage. The tunneling current is held constant throughout the scan. Changes of only 0.1 nm in the separation distance cause an order of magnitude difference in the tunneling current. The

interaction is between single atoms in the sample and tip, giving STM remarkably high lateral resolution.

Keysight STM tips are pre-cut or chemically etched lengths of 0.25 mm OD, 80 % platinum - 20 % iridium wire. If the wire tip is damaged it can be trimmed and used again. Using tips coated in insulating wax, STM can also be performed in fluid.

The Keysight multi-purpose scanner, when equipped with an STM nose assembly (Figure 92), can be used for STM. The nose assembly is available with three different preamplifiers for varying sensitivity (Table 3).



Figure 92 STM nose assembly

Table 3 STM nose assembly and scanner sensitivities

Color	Red	Blue	Green
Sensitivity	10 nA/V	1 nA/V	0.1 nA/V
Bandwidth	20 kHz	6.3 kHz	2 kHz
Test Resistor	100 M Ω	1 G Ω	10 G Ω

A dedicated STM scanner (Figure 93) provides lower current operation and higher resolution. The scanner is available with three preamplifier options for varying sensitivity (see Table 3). The color-coded preamp,

located beneath the tip, can be field-replaced to adjust the sensitivity if necessary.



Figure 93 STM scanner

STM Gain and Preamp Settings

The STM scanner has two amplifier stages: gain stage and preamp stage. Each stage can be set to different values as described below.

Gain Stage Settings

As shown in [Figure 94](#), the Gain switch set to the right side position gives a gain of 1. When the switch is set to the left side position, it gives a gain of 10.

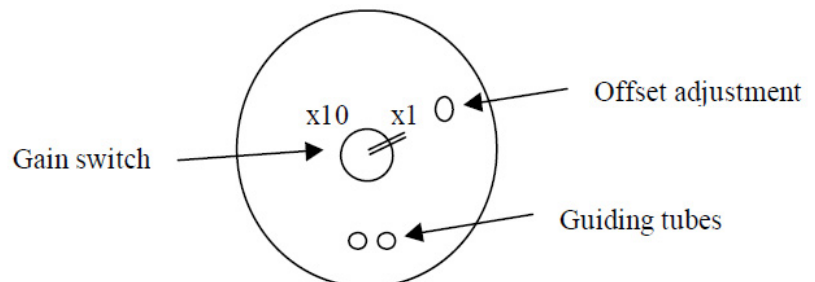


Figure 94 Gain switch settings for STM scanner

Preamp Stage Settings

The preamp stage uses a fixed preamp board module as shown in [Figure 95](#). It can be replaced with different sensitivity settings as noted in [Table 3](#). The standard scanner has the module with 10 nA/V sensitivity. The optional modules are 1 nA/V and 0.1 nA/V. Other values are available upon request.

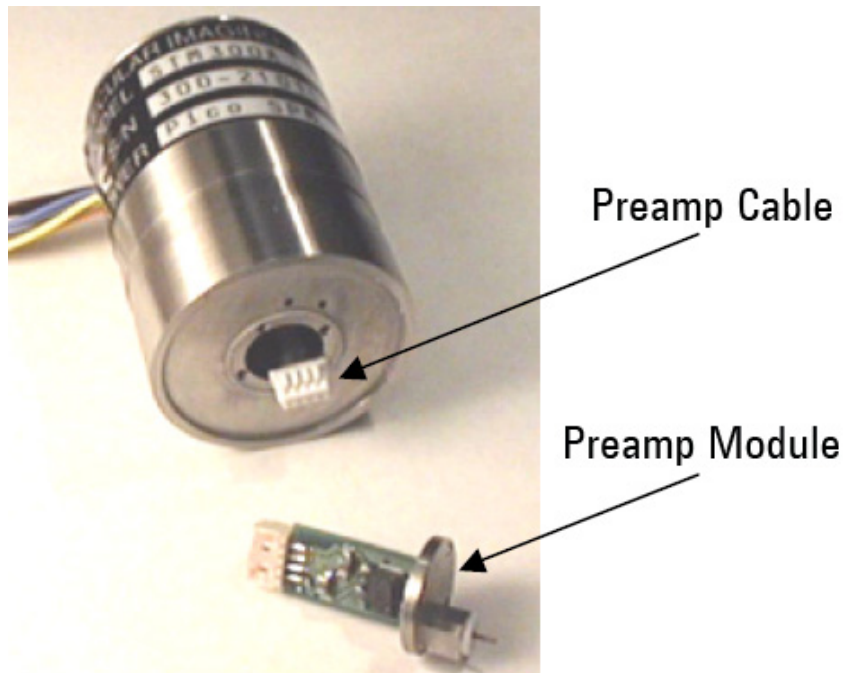


Figure 95 STM scanner preamp cable and module

To exchange these modules, please follow the procedure described below.

CAUTION

Do not pull the cable while unplugging the module. Hold the 4-pin connector at the end of the cable firmly, then unplug the module as shown in [Figure 96](#).

- 1 Remove the four screws on the tip holder piece.
- 2 Pull out the preamp module carefully.
- 3 Hold on to the 4-pin connector and unplug the module from the cable.
- 4 Replace the module and plug it back onto the 4-pin connector.

- 5 Tighten the module back onto the scanner.

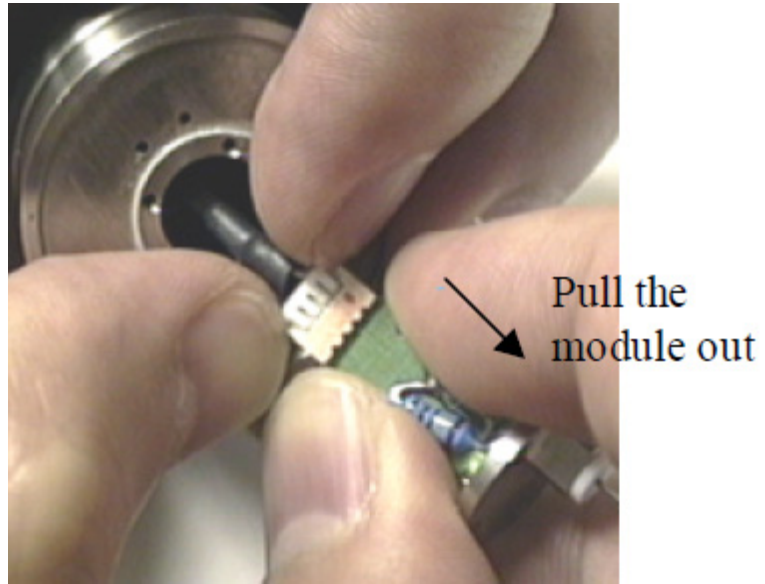


Figure 96 Hold the connector while unplugging the preamp module

Final Sensitivity Calculation

To calculate the final sensitivity, use this equation:

$$\text{Final Sensitivity} = (\text{PreampSensitivity})/(\text{Gain})$$

For example, if the Gain switch is set to 10 and the module has a 10 nA/V preamp, the final sensitivity will be 1 nA/V.

STM Imaging Procedure

The procedure for STM imaging is as follows:

- 1 If you are using the multi-purpose scanner, insert the nose assembly into the scanner (see [Chapter 4](#) for details).
- 2 Insert a tip into the nose assembly or scanner. Grasp the tip with a tweezers, then insert it into the hollow tube until it protrudes approximately 2 mm ([Figure 97](#)).

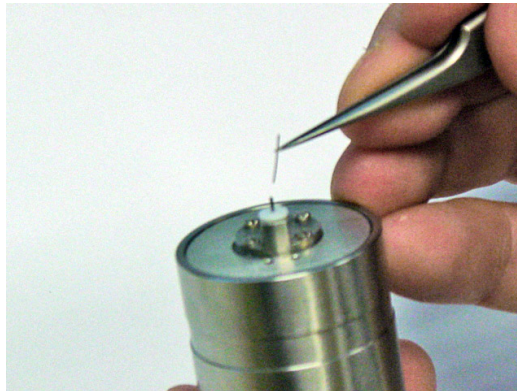


Figure 97 Inserting STM tip wire in scanner

- 3 Place the scanner in the scanner bracket on the microscope.
- 4 Prepare the sample and place it on a sample plate. The sample must be electrically isolated from the sample plate. The particular mounting arrangement will depend on the sample type and size.
- 5 Attach an electrode from the sample plate to the sample. Lift the spring-loaded electrode clip on the sample plate and insert the

electrode under it (Figure 98). Connect the electrode to the sample, ensuring good contact.

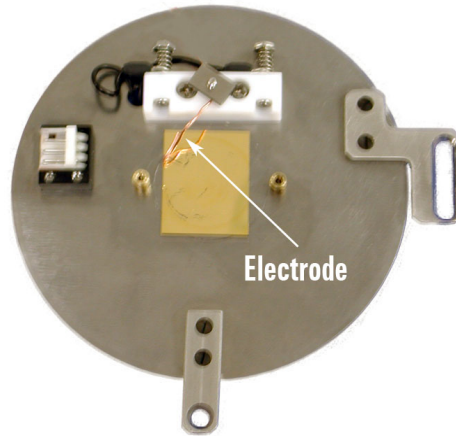


Figure 98 Sample on plate with electrode attached

- 6 Place the sample plate on the microscope.
- 7 Plug the 3-pin EC connector of the EC/MAC cable into the 3-pin socket on the sample plate. Plug the other end of the cable into the EC/MAC socket on the microscope.

NOTE

The sample plate cable can transfer low levels of vibration to the sample. During very high resolution imaging this can affect images quality. We recommend first plugging the sample plate cable to the flexible 3-wire umbilical included with the sample plate. The umbilical should then be plugged in to the microscope base. The umbilical's individual wires tend to reduce the transfer of vibration.

- 8 In PicoView, choose **Mode > STM**.
- 9 In the Servo window enter the Bias Voltage (Figure 99). Typical values are **50-200 millivolts** (0.05-0.200 V). A positive bias indicates current flow *from* the tip *to* the sample, and vice versa for negative bias.

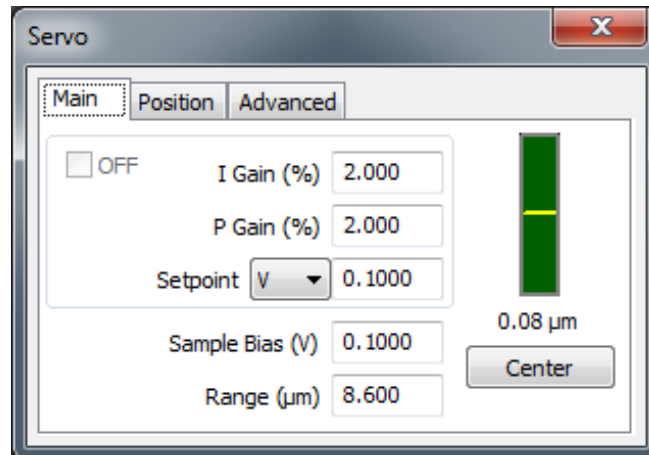


Figure 99 Servo window settings for STM imaging

- 10 Enter the Setpoint current, in nanoamps, that the system will try to hold constant during scanning. A typical setting is **1-2 nA**.
- 11 Enter the **I** and **P** gains for the z-servo, which will dictate how quickly the system will adjust to changes in tunneling current. Typical values are **1-2 %** for both gains.
- 12 In the Realtime Images window choose to display images for **Current** and **Topography**.
- 13 In the Scan and Motor window set the scan size, speed and offsets. A scan **Speed** of **1 In/s** is a good starting value.
- 14 Using the **Close** switch on the HEB, raise the sample until the tip is close to, but not touching, the scanner. The video system is not useful in STM as the tip is essentially vertical, so view the tip from the side of the microscope and bring it as close to the sample as you can. Be certain to not drive the tip into the sample. To be safe you can make the approach length longer, which will just add a little time to the approach.
- 15 Click the **Approach** button in PicoView's toolbar. The scanner will lower until the Setpoint current is reached.
- 16 For lowest current operation, once engaged reduce the Setpoint value until the indicator in the Servo window changes from green to red. Then increase the Setpoint until the indicator in the Servo window just turns green. For rougher surfaces you may need to increase the setpoint current slightly more.
- 17 In the Scan and Motor window, click the **Up** or **Down** arrows to begin the scan.

Current Sensing AFM (CSAFM)

In Current Sensing AFM (CSAFM) an ultra-sharp AFM cantilever, coated with conducting film, probes the conductivity and topography of the sample surface. CSAFM requires a special 10° nose cone containing a pre-amp. A bias voltage is applied to the sample while the cantilever is kept at virtual ground (Figure 100). As in Contact Mode, the tip force is held constant throughout the scan. The current is used to construct the Conductivity image.

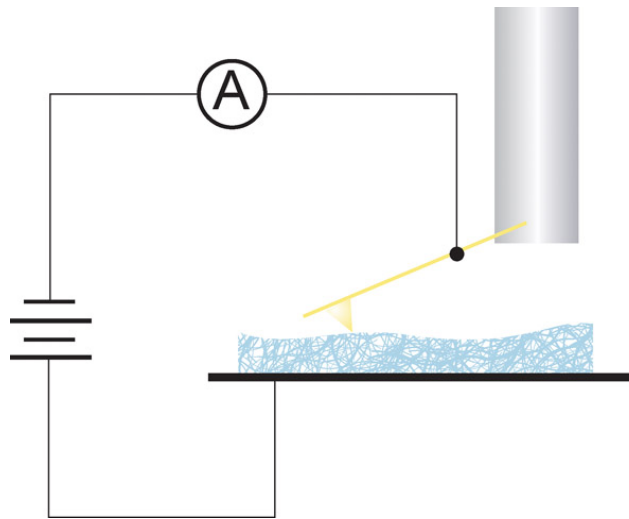


Figure 100 CSAFM schematic

CSAFM is useful for locating defects in thin films, for molecular recognition studies, and for resolving electronic and ionic processes across cell membranes. It has proven useful in joint I/V spectroscopy and contact force experiments as well as contact potential studies.

CSAFM imaging can be used in an ambient environment or under temperature or environmental control. However, as surface contamination (especially a moisture layer on the sample surface) can reduce the clarity of imaging, it is strongly recommended that CSAFM be completed in a controlled, low humidity environment.

The Keysight multi-purpose scanner can be used with the CSAFM nose assembly (Figure 101) for CSAFM imaging. The nose assembly includes one of three color-coded preamps for varying sensitivity:

10 nA/V (red), 1 nA/V (blue) or 0.1 nA/V (green). See [Table 3](#) on page 133 for more details.



Figure 101 CSAFM nose assembly

Platinum-coated, conductive tips are required for CSAFM imaging. Because an electrode must be attached to the sample, a sample plate is also required.

CAUTION

The CSAFM nose assembly cannot be used for imaging in liquid.

To image in CSAFM Mode:

- 1 Begin with the steps you learned in [Chapter 4](#):
 - a Insert the nose assembly into the scanner.
 - b Load a probe into the nose assembly.
 - c Place the scanner in the microscope base and connect its cables.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
- 2 Prepare the sample and place it on a sample plate. The sample must be electrically isolated from the sample plate. The particular mounting arrangement will depend on the sample type and size.
- 3 Attach an electrode from the sample plate to the sample. A length of copper wire works well as the electrode. Lift the spring-loaded electrode clip on the sample plate and insert the electrode under it ([Figure 98](#) on page 138). Connect the electrode to the sample. Check

the continuity between the working electrode contact and sample to ensure that a proper connection is achieved.

- 4 Place the sample plate on the microscope.
- 5 Plug the 3-pin EC connector of the EC/MAC cable into the 3-pin socket on the sample plate. Plug the other end of the cable into the EC/MAC socket on the microscope.

NOTE

The sample plate cable can transfer low levels of vibration to the sample. During very high resolution imaging this can affect resolution. We recommend first plugging the sample plate cable to the 3-wire umbilical included with the sample plate. The umbilical should then be plugged in to the microscope base. The umbilical's individual wires tend to reduce the transfer of vibration.

- 6 In PicoView, choose **Mode > CSAFM**.
- 7 In the Servo window enter the Bias Voltage. Typical values are **50-200 millivolts** (0.05-0.200 V).
- 8 Using a voltmeter, check the potential between the working electrode contact and ground (the exposed metal of the DB44 connector on the microscope is a good ground point). The bias should be the same as that entered in the Servo window. If it is not, you may need to adjust the controller calibration (see the PicoView software user guide for additional information).
- 9 Enter a **Setpoint** value that is slightly more positive than the current Deflection reading (on the HEB front panel or PicoView's Laser Alignment window).
- 10 Enter the **I** and **P** gains for the z-servo, which will dictate how quickly the system will adjust to changes in tip deflection. A typical starting value is **10 %** for both gains.
- 11 In the Realtime Images window choose to display images for **CSAFM/Aux BNC, Deflection and Topography**.
- 12 In the Scan and Motor window's **Scan** tab, enter:
 - a Scan Speed of **1-2** ln/s.
 - b Resolution of **256**.
 - c Scan **Size** (in microns).
 - d **X Offset** and/or **Y Offset** values to set the location of the scan center.
- 13 Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.

- 14 Watching the video system, bring the tip and sample very close to contact:
 - a Adjust the focus and location of the video such that the tip is in sharp focus.
 - b Lower the focal plane just slightly below the tip by turning the Focus control toward you until the tip is slightly out of focus.
 - c Now, using the **Close** switch on the HEB, raise the sample until the sample comes nearly into focus. The tip should now be just above the sample surface.

CAUTION

Raise the sample slowly and carefully to avoid collision with the sample. Hard contact between the tip and the sample can damage either or both.

- 15 Click the **Approach** button in PicoView's toolbar. The scanner will be lowered until the Setpoint deflection voltage is reached.
- 16 In the Servo window, make the **Setpoint** more negative until the tip leaves contact with the sample—the indicator in the Servo window will change from green to red. Raise the Setpoint again until the Servo window indicator just turns green.
- 17 In the Scan and Motor window, click the **Up** or **Down** arrows to begin the scan.

During the scan, the system will maintain a constant force on the tip, and Deflection and Topography will be imaged as in Contact Mode. The tip itself will remain at virtual ground as the bias is applied to the sample. The current signal will be positive when the sample surface is biased positively. The CSAFM image will show highly conductive regions as “high” features.

The amplitude of the current signal is strongly dependent upon the condition of the cantilever tip and sample surface, as well as the force applied to the surface. Using known good tips, a controlled environment and low tip force will improve imaging.

Lateral Force Microscopy (LFM)

Lateral Force Microscopy is a derivative of Contact Mode. During a typical scan, the cantilever twists in the scan direction as well as deflecting in the vertical axis. The detector senses change in the cantilever's twisting motion and outputs it as the lateral deflection (Friction) signal.

Changes in lateral force on the tip can be caused either by changes in frictional properties across the sample or by variations in topography. The Friction signal will therefore be a convolution of these two components. To differentiate friction from topography, two LFM images are typically captured side-by-side. One image is constructed from the Friction signal during each trace of the raster scan, and the other from the Friction signal during retrace. One image can then be inverted and subtracted from the other to reduce the topographic artifacts, leaving primarily the effects of friction.

To image in LFM Mode, follow the procedure for Contact Mode given in [Chapter 5](#). In the Realtime Images window, choose to display two Friction images, selecting **Trace** for one and **Retrace** for the other ([Figure 102](#)).



Figure 102 Display Trace and Retrace Friction images

NOTE

It is important in LFM that the LFM signal on the Head Electronics Box be carefully set as close to zero as possible.

Dynamic Lateral Force Microscopy (DLFM)

In Dynamic Lateral Force Microscopy (DLFM), a lead zirconate titanate (PZT) ceramic element in the nose cone oscillates the tip parallel to the sample surface, in the direction of the scan (as opposed to perpendicular oscillation as in AC Mode). Cantilever deflection is mapped to give topography, as in contact mode. Changes in the amplitude and phase of the lateral oscillation are imaged. DLFM is very sensitive to changes in surface properties such as friction and adhesion, and as such it is particularly useful for polymer studies.

DLFM requires a DLFM nose assembly and any sample plate. Force Modulation cantilevers are recommended, with a resonance frequency in the 70-80 kHz range. Some experimentation with stiffer or softer probes may be required to achieve satisfactory imaging. A MAC Mode or MAC III controller is also required to drive the lateral oscillation.

CAUTION

Electrical elements of the DLFM nose assembly are exposed. Therefore, DLFM should **never** be performed in liquid.

With the MAC Mode controller the following cables must be added:

- Connect a BNC cable from the **Phase** output of the MAC Mode controller to the **Aux In** of AFM Controller.
- Connect a BNC cable from the **Amplitude** output of the MAC Mode controller to the **Aux BNC** on the Head Electronics Box.

For the MAC III controller these connections are made in software.

To image in DLFM Mode:

- 1 Begin with the steps you learned in [Chapter 4](#):
 - a Insert the nose assembly into the scanner.
 - b Load a probe into the nose assembly.
 - c Place the scanner in the microscope base and connect its cables.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
- 2 In PicoView choose **Mode > DLFM**.
- 3 Choose **Controls > AC Mode** to open the ACAFM Controls window.
- 4 Set the Drive Mechanism to **AAC**.
- 5 Set the Drive % to **10 %**.

- 6 Bring the tip close to the sample:
 - a Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.
 - b Focus the cantilever in the video window.
 - c Turn the video system focus knob toward you such that the tip goes just out of focus.
 - d Press the **Close** switch to raise the sample until both the tip and sample are in focus (i.e., they are nearly touching).
- 7 In the Servo window, enter a **Setpoint** value slightly more positive than the current Deflection reading. This sets the force on the tip that will represent “contact” both during approach and during the scan.
- 8 Click the **Approach** button in PicoView’s toolbar. The system will raise the sample until the deflection reaches the Stop At value.
- 9 After approach, a scan may be performed to check for a region of interest and to optimize the scanning parameters. When the area of interest has been located, stop the scan.
- 10 Set the oscillation frequency for the cantilever:
 - a Choose **Controls > Advanced > AC Mode**. Select **Friction** as the Input. This will cause the lateral signal from the detector to be used for tuning the resonance of the cantilever, rather than the deflection signal.
 - b Choose **Controls > AC Mode Tune** to open the AC Tune window.
 - c In the Manual Tune (bottom section) of the window, enter appropriate **Start** (kHz) and **End** (kHz) frequencies. The frequency range should encompass the possible resonance frequency of the cantilever. The frequencies are generally in the 20-50 kHz range.
 - d Click the **Manual Tune** button.
 - The system will perform a single frequency sweep from the Start to the End frequency.
 - Note that the frequency can be selected by moving the vertical dashed bar in the frequency plot.
 - e Experimentation will probably be required to determine the best frequency for each tip and sample combination but a good starting point is a frequency that produces the largest deflection.
- 11 In the Servo window, set the I Gain and P Gain to **5 %**.

- 12 In the Scan and Motor window's **Scan** tab, enter:
 - a Scan Speed of **1-2** ln/s.
 - b Resolution of **256**.
 - c Scan **Size** (in microns).
 - d **X Offset** and/or **Y Offset** values to set the center of the scan.
- 13 In the Realtime Images window, choose to display **Topography**, **CSAFM/BNC Aux** (Phase) and **HEB Aux** (Amplitude). If using a MAC III controller, select **Aux 1** and **Aux 2**.
- 14 In the Scan and Motor window, click the **Up** blue arrow to initiate a scan starting at the bottom of the grid. Click the **Down** blue arrow to initiate the scan from the top down. The image maps will begin to be rendered in the Realtime images window.

Force Modulation Microscopy (FMM)

Force Modulation is another derivative of Contact Mode, with similarities to AC Mode as well. In FM Mode, an additional 20-50 kHz oscillation is applied to the cantilever. The amplitude and phase of oscillation will change depending upon the modulus of the surface at any given point.

The multi-purpose scanner and AAC nose assembly are used for FM Mode. A specific Force Modulation cantilever is available through Keysight; however, the best choice of cantilever is often experimentally determined. A MAC Mode or MAC III controller is also required.

With the MAC Mode controller the following cables must be added:

- Connect a BNC cable from the **Phase** output of the MAC Mode controller to the **Aux In** of AFM Controller.
- Connect a BNC cable from the **Amplitude** output of the MAC Mode controller to the **Aux BNC** on the Head Electronics Box.

For the MAC III controller these connections are made in software.

To image in Force Modulation Mode:

- 1 First follow the steps from [Chapter 4](#)
 - a Insert the nose assembly into the scanner.
 - b Insert a probe into the nose assembly.
 - c Place the scanner in the microscope base.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
 - f Prepare the sample and place it on the sample plate.
 - g Configure the cable connections for MAC as described in [Figure 207](#) on page 307.
 - h Adjust the video system to focus on the cantilever.
- 2 Choose **Controls > Setup > Components**, then select the **Serial Port AC Mode Controller** check box. The system will now use the signal from the MAC (or MAC III) controller.
- 3 In PicoView choose **Mode > Contact**. Or, if you are using a MAC III controller, choose **Mode > Force Modulation**.
- 4 Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.
- 5 Use the video system to bring the tip and sample close to contact:
 - a Bring the cantilever into sharp focus.
 - b Lower the focal plane just slightly below the tip by turning the Focus control toward you until the tip is slightly out of focus.
 - c Using the **Close** switch on the HEB, raise the sample until the sample and tip both come nearly into focus. The tip should now be just above the sample surface.
- 6 Locate the area of interest on the sample by performing a scan.
- 7 In PicoView's Servo window, enter a **Setpoint** value slightly greater than the current Deflection reading (from the HEB front panel or PicoView's Laser Alignment window).
- 8 Click the **Approach** button. The system will raise the sample until the deflection reaches the Setpoint value.
- 9 Now set up the additional AC oscillation:
 - a Choose **Controls > AC Mode** to open the ACAFM Controls window.
 - b Set the Drive Mechanism to **AAC**.
 - c Set the Drive % to 10 %.
 - d Set the Frequency to 20-50 kHz.

- 10 In the Servo window set the I Gain and P Gain to **5 %**.
- 11 In the Scan and Motor window's **Scan** tab, enter:
 - a Scan Speed of **1-2** ln/s.
 - b Resolution of **256**.
 - c Scan **Size** (in microns).
 - d **X Offset** and/or **Y Offset** values to set the center of the scan.
- 12 In the Realtime Images window, display three images for **Topography**, **CSAFM/Aux BNC** (the Phase signal via the MAC controller), and **HEB Aux** (the Amplitude signal via the HEB). If using a MAC III controller, select **Aux 1** and **Aux 2**.
- 13 In the Scan and Motor window, click the **Up** or **Down** blue arrows to initiate a scan.

Magnetic Force Microscopy

Magnetic Force Microscopy (MFM) is a derivative mode of Contact Mode imaging, AC Mode, and also of **Dynamic Lateral Force Microscopy (DLFM)**.

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

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

Since the tip oscillates with a non-zero amplitude, the distance between the tip and the sample changes during each oscillation cycle, and with it so does the strength of the stray magnetic field and the tip-sample magnetic interaction: The gradient of the magnetic field above the sample surface leads to a gradient of the tip-sample force, which then alters the cantilever's motion. It is this force gradient that the MFM mode detects and maps into the image, using **Interleave** scanning. At a given drive frequency, the phase and the amplitude of the cantilever oscillations change. These changes can be recorded pixel by pixel to generate an MFM image. When the phase signal is mapped in this way, an MFM phase image is created.

Interleave

Some modes require two scans of the surface. The cantilever scans once in the X direction (trace and retrace), measuring topography, then raises the cantilever above the surface and scans the same line again (trace and retrace). This interlacing of scans on a line-by-line basis is called an

Interleave scan. The distance from the surface for the interleave scan is determined by the Interleave Pull Away and Stop At parameters.

The topography and the MFM force gradient can be mapped simultaneously and displayed side-by-side to highlight correlation between the magnetic response and topography. For a typical MFM-type cantilever with a force constant in the range of 3 N/m and a resonance frequency of about 75 kHz, the following setup should be utilized, beginning with a standard topography image setup:

- 1 To image in MFM Mode, first follow the steps from **Chapter 4**:
 - a Insert the nose assembly into the scanner.
 - b Insert an MFM probe into the nose assembly.
 - c Place the scanner in the microscope base.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
- 2 Prepare the sample and place it on a sample plate.
- 3 Choose **Mode > AC Mode**.
- 4 Choose **Controls > AC Mode Tune** to open the AC Tune window.
- 5 Tune the drive signal to the resonance frequency of the cantilever:
 - a In the AC Tune window, enter the Start and End frequencies (in kHz) for the tuning sweep, typically **20-120 kHz**.
 - b Set the Peak Amplitude to **3.5 to 5.0** volts.
 - c Enter an Off Peak value to offset the oscillation frequency from the cantilever's resonance frequency. A typical value is **-0.300 kHz**. Using that setting, if the resonance frequency is 72.539 kHz, the drive frequency in the AC Tune window should be set by the Auto Tune process to 72.239 kHz.
 - d Click the **Auto Tune** button. The system will sweep through the range of frequencies, locating the peak oscillation amplitude within the range. This should correspond to the resonant frequency of the cantilever. The AC signal oscillation frequency will be set to the resonant frequency, offset by the Off Peak parameter in the AC Tune window.
- 6 Bring the tip close to contact with the sample:
 - a Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.
 - b Focus the cantilever in the video window.
 - c Turn the video system focus knob toward you such that the tip goes just out of focus (the focal plane is just below the tip now).
 - d Repeat this iteratively (steps a-c) until both the tip and sample are in focus (i.e., they are nearly touching).

- 7 In the Realtime Images window (Figure 103), choose the two images to display **Topography** (input channel circled in red) with the **Main** scan mode (circled in blue) for one and **Phase** with the **Interleave** scan mode for the other. Set both images to **Retrace**.

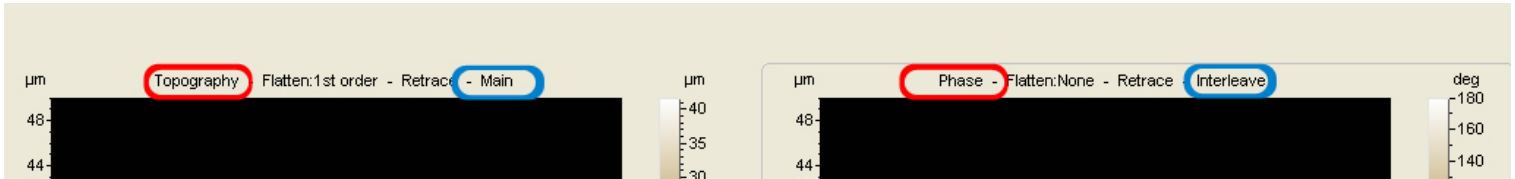


Figure 103 Realtime images settings for MFM with input channels set to Topography and Phase, scan modes set for Main and Interleave, respectively.

NOTE

It is sometimes useful to open the Trace/Retrace window while optimizing imaging parameters. The Trace/Retrace window inputs are NOT dependent on the settings in the Realtime Imaging window. The scan modes should be set to Main in the Trace/Retrace window when Interleave scanning is completed if changed during MFM imaging.

Set up the Interleave parameters. In the Scan and Motor window, click on the **Interleave** tab (Figure 104).

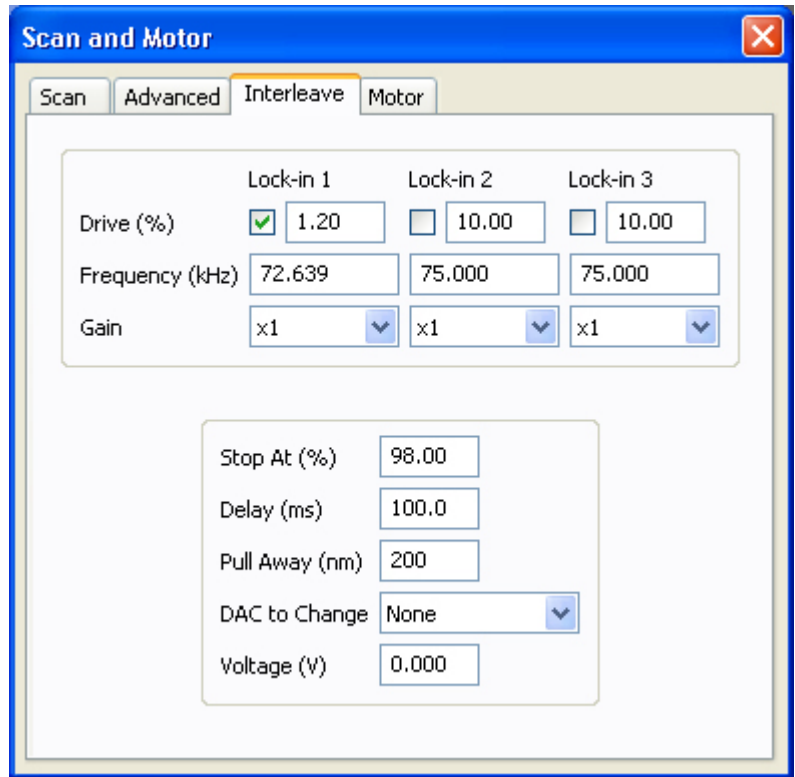


Figure 104 Interleave tab used for MFM imaging

- 1 Select the **Drive (%) Lock-in 1** check box.
- 2 Set the Lock-in 1 Frequency to **0.100 kHz** above the cantilever resonance frequency. In the case above (step 5) where the cantilever resonance frequency was 72.539 kHz, the Interleave Frequency would be set to 72.639 kHz.

- 3 Set the Interleave Drive (%) so the amplitude is about 1/5 the free amplitude used for the topography scan:
 - a Choose **Controls** > **AC Mode** to open the ACAFM Controls dialog box.
 - b Adjust the **Drive (%)** to determine the value needed to reduce the amplitude for the Interleave setting of 1/5 free amplitude.
 - c Enter the Drive (%) value from the ACAFM Controls dialog box into the Interleave Drive (%) (**Figure 104**) for MFM imaging.
 - d Re-set the Drive (%) in the the ACAFM Controls dialog box to its original value.
- 4 Set the Interleave Gain to **x1** or **x2**. Higher gains may cause the servo to oscillate.
- 5 Set the Interleave Stop At value above **95 %**.

Setting the Stop At value too low can cause the tip-sample interactions to dampen out the MFM data. Setting it too high can cause the sensitivity of the tip to drop.

- 6 Set the Delay to **100 ms**.
- 7 Set the Pull Away to **200 nm**.
- 8 DAC to Change should be set to **None**.

The Delay parameter is a time delay prior to the beginning of the sweep for the interleave channel. After clicking the Start button, the tip will move to the Start location, then the system will pause the Delay time, allowing the system to stabilize.

The Pull Away parameter sets the distance the tip is pulled away from the surface to initiate the changes in the drive frequency and the drive amplitude. Adjust Pull Away from between 100 and 200 nm to optimize the image.

Begin imaging:

- 1 Initiate an approach:
 - a In the Scan and Motor window, click the **Motor** tab.
 - b Set the Stop At (%) to specify the percentage of total oscillation that represents “contact,” typically **85-90 %**.
 - c Click the **Approach** button in PicoView’s toolbar. The system will raise the sample until the amplitude is damped to the Stop At percentage.
- 2 In the Servo window, set the I Gain and P Gain to **5 %**. These gains dictate how quickly the system will react to changes in amplitude.
- 3 In the Scan and Motor window’s **Scan** tab, enter:
 - a Scan Speed of **1-2** ln/s.
 - b Resolution of **512**.
 - c Scan Size (in microns).
 - d X Offset and/or Y Offset values to set the center of the scan.
- 4 In the Scan and Motor window, switch to the Scan tab and click the **Up** or **Down** blue arrow to initiate a scan. The phase map will show changes in the magnetic force during interleave scanning.
- 5 In the Servo window Main tab and the Scan and Motor window Scan tab, adjust the Gain, Scan Speed, Resolution, etc., to optimize the topography image.
- 6 In the Interleave tab, adjust the Gain, Stop At and Pull Away parameters to optimize the MFM phase image.

Electrostatic Force Microscopy (EFM)

Electrostatic Force Microscopy (EFM) is a *qualitative* method for examining changes in the intrinsic or applied electrostatic field of a sample surface. EFM is a derivative of AAC Mode, using a conductive tip. A bias voltage is applied to the sample, allowing local static charge domains and charge carrier density to be measured. EFM has proven useful for examining fuel cells, solar cells, and for troubleshooting semiconductor circuits to locate leaks and shorts.

EFM Mode requires a MAC III controller to provide the drive signals. Lock-in 1 is used to drive the cantilever. The input to Lock-in 1 is the amplitude of the cantilever deflection at a specific frequency. Lock-in 2 operates at a different frequency, providing the AC bias, also with the Deflection channel as its input.

An AC nose assembly and any sample plate with an electrode connection are required. Conductive EFM tips with a resonance of approximately 60 kHz are required.

The phase of the Lock-in 2 signal changes in response to changes in the electric field as the tip passes over the surface. The real component of the phase (X Component 2) and the total phase can both be mapped. A standard topography image can be collected at the same time. The two images can then be displayed side-by-side to highlight correlation between the electrostatic response and topography.

- 1 To image in EFM Mode, first follow the steps from [Chapter 4](#):
 - a Insert the nose assembly into the scanner.
 - b Insert a probe into the nose assembly.
 - c Place the scanner in the microscope base.
 - d Align the laser on the cantilever.
 - e Insert and align the detector.
- 2 Prepare the sample and place it on a sample plate. The sample must be electrically isolated from the sample plate.
- 3 Attach a conductor (typically a stiff wire) from the working electrode to the sample. Lift the spring-loaded electrode clip on the sample plate and insert the conductor under it. Connect the conductor to the sample. Check the continuity between the working electrode contact and sample to ensure a good connection.
- 4 Plug the 3-pin EC connector of the EC/MAC cable into the 3-pin socket on the sample plate. Plug the other end of the cable into the EC/MAC socket on the microscope.
- 5 Choose **Mode > EFM**.

- 6 Choose **Controls > Advanced > AC Mode**. The EFM Controls window will open (Figure 105).

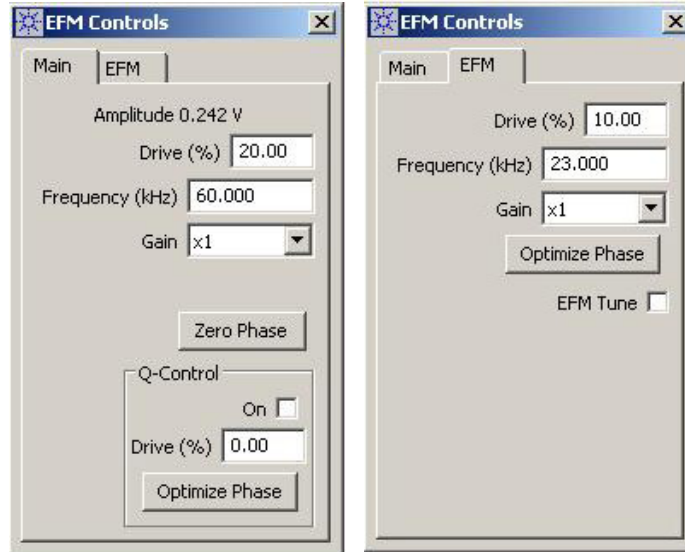


Figure 105 EFM Controls window

- 7 In the Main tab, set up the Lock-in 1 AC signal which drives the cantilever oscillation:
- Set the Drive % to 10 %.
 - Set the Gain to **x1** (the amplitude times 1).
- 8 Choose **Controls > AC Mode Tune** to open the AC Tune window.
- 9 Tune the drive signal to the resonance frequency of the cantilever:
- In the AC Tune window, enter the **Start** and **End** frequencies (in kHz) for the tuning sweep, typically 20-120 kHz.
 - Set the Peak Amplitude to **2.5** volts.
 - Enter an **Off Peak** value to offset the oscillation frequency from the cantilever's resonance frequency. A typical value is **-0.100 kHz**.
 - Click the **Auto Tune** button. The system will sweep through the range of frequencies, locating the peak oscillation amplitude within the range. The AC signal oscillation will be set to this value plus the specified Offset.

NOTE

Q Control can be used with EFM Mode to improve the resonance peak and image quality. See [Chapter 6](#) for more on Q-Control.

- 10 Bring the tip close to contact with the sample:
 - a Press the **Close** switch on the HEB to raise the sample until the tip is close to, but not touching, the sample.
 - b Focus the cantilever in the video window.
 - c Turn the video system focus knob toward you such that the tip goes just out of focus (the focal plane is just below the tip now).
 - d Press the **Close** switch to raise the sample until both the tip and sample are in focus (i.e., they are nearly touching).
- 11 Now initiate an approach:
 - a In the Scan and Motor window, click the **Motor** tab.
 - b Set the Stop At (%) to specify the percentage of total oscillation that represents “contact,” typically **90-95 %**.
 - c Click the **Approach** button in PicoView’s toolbar. The system will raise the sample until the amplitude is damped to the Stop At percentage.
- 12 On the EFM tab, you will now set up the MAC III controller’s second lock-in to provide the AC bias. Use the AC Tune window to verify that this signal is at a frequency that does not add unwanted tip responses.
 - a In the Servo window, note the **Setpoint** value. Change the Setpoint to **10 V** to move the tip several microns above the sample.
 - b In the EFM tab, set the Drive % to **10 %**. The Drive % value is somewhat experimental; a higher value will improve image contrast but, beyond a point, it will add noise.
 - c Enter a **Frequency** that is smaller than, and not an even factor of, the Lock-in 1 signal (from the Main tab). For example, if the Lock-in 1 Frequency is 60 kHz choose a frequency other than 30, 15, or 7.5 kHz.
 - d Set the **Gain**, which multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve signal-to-noise ratio for a small signal. Ensure that the gain will not result in an

amplitude exceeding 10 V, beyond which the signal will be clipped. The default value is **x1** (the amplitude times 1).

- e Select the **EFM Tune** check box.
 - f In the AC Tune window, set the **Start** and **End** frequencies for the EFM tune sweep. Use a wide range centered on the Lock-in 2 frequency.
 - g Click the **Manual Tune** button. The system will sweep Lock-in 2 through the range of frequencies, displaying any peak oscillation amplitude within the range.
 - h Adjust the **Frequency** in the EFM tab if it falls close to one of these peak frequencies.
 - i In the Servo window, return the **Setpoint** to its original value.
- 13 On the EFM tab, clear the **EFM Tune** check box.
 - 14 On the EFM Controls Main tab, click the **Zero Phase** button to set the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
 - 15 On the EFM tab, click **Optimize Phase**. This will shift the phase of the Lock-in 2 signal to maximize the X component of phase.
 - 16 In the Servo window set the I Gain and P Gain to **5 %**. These gains dictate how quickly the system will react to changes in amplitude.
 - 17 In the Scan and Motor window's **Scan** tab, enter:
 - a Scan Speed of **1-2** ln/s.
 - b Resolution of **256**.
 - c Scan Size (in microns).
 - d **X Offset** and/or **Y Offset** values to set the center of the scan.
 - 18 In the Realtime Images window, choose to display **Topography** and **Aux 1** (the X component of the phase signal which will be mapped to make the EFM image).
 - 19 In the Scan and Motor window, click the **Up** or **Down** blue arrow to initiate a scan. The Aux 1 map will show changes in the electrostatic force as they differ from the force at the touch-down location.
 - 20 Adjust the Gain, Scan Speed, Resolution, etc., to optimize the topography image.

For more on advanced options for EFM Mode see [Chapter 11](#).

Kelvin Force Microscopy (KFM)

Kelvin Force Microscopy (KFM) is similar to EFM. An additional feedback loop applies a DC bias to the tip to counteract deflection due to the surface electrostatic force. The output from this feedback loop provides a *quantitative* analysis of changes in the applied or intrinsic electrostatic field of the sample.

As in EFM Mode, KFM requires conductive tips, a sample plate with electrode connection, an AC nose assembly, and a MAC III controller to provide the drive signals. Lock-in 1 is used to drive the cantilever. Lock-in 2 provides an AC bias. The MAC III internal servo drive provides the DC bias to counteract tip deflection.

The procedure for imaging in KFM Mode is the same as that for EFM Mode, with the additional step of setting up the DC bias servo. This should be completed after approach:

- 1 Choose **Controls > Advanced > AC Mode**, then click the **Other** tab (Figure 106).

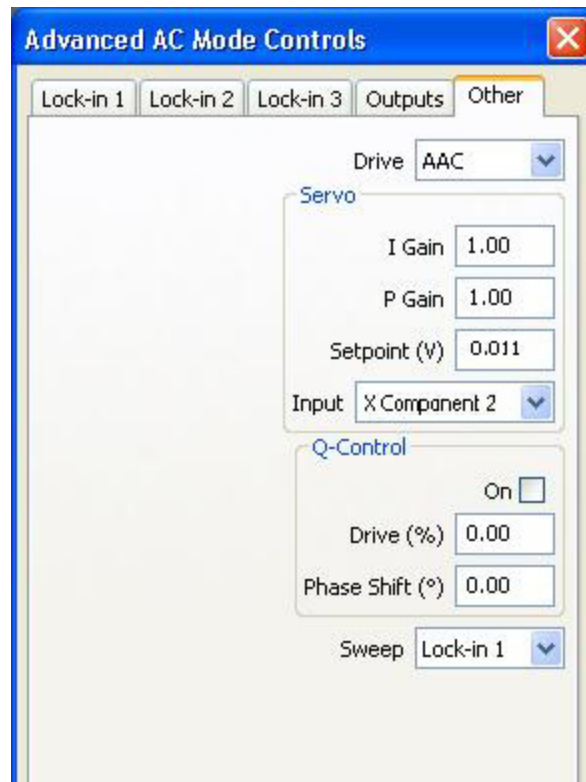


Figure 106 Advanced AC Mode Controls Other tab

- 2 Set the **I** and **P** gains for the Servo to **1 %**.

- 3 In the Realtime Images window, choose to display images for **Topography**, **Phase** and **SP** (the output from the servo).
- 4 Choose **Controls >Spectroscopy** to open the Spectroscopy window.
- 5 Confirm that **Amplitude vs Distance** is the plot type.
- 6 Open a second buffer and set the Display Option to **SP**.
- 7 On the Lock-in 2 tab of the Advanced AC Mode Controls window, verify that the **From Servo** check box is cleared.
- 8 To prevent damage to the tip during the Amplitude vs Distance cycle, the total motion of the piezo needs to be reduced so that it does not contact the sample surface. Note the position of the piezo in the Servo window indicator. Enter a slightly more positive value for the **End** parameter in the Spectroscopy window.
- 9 Click on the blue triangle to begin the piezo movement and observe the SP response in the Spectroscopy window.
- 10 In the Advanced AC Mode Controls window Other tab, increase the **Setpoint** value in small increments until the SP vs Distance plot is as horizontal as possible. Hold down the **Shift** and **Ctrl** keys while pressing the **Up** arrow on the keyboard to increment the amount by 0.001. The traces will probably be noisy, but make them as horizontal as possible. This adjustment has the largest effect on the output of the SP servo and typically has a value of less than 0.05. The servo values should now be optimized.
- 11 Stop the Spectroscopy.
- 12 On the Lock-in 2 tab, select the **From Servo** check box.

For more on advanced options for KFM Mode see [Chapter 11](#).

Force Spectroscopy

Force Spectroscopy is an AFM-based technique to measure, and sometimes control, the polarity and strength of the interaction between the AFM tip and the sample. Although the tip-sample interaction may be studied in terms of the energy, the quantity that is measured first is always the tip-sample force. Unlike imaging, force spectroscopy is performed mostly when the servo feedback loop is deactivated. In force spectroscopy, the cantilever-tip assembly acts as a force sensor, as shown schematically in [Figure 107](#).

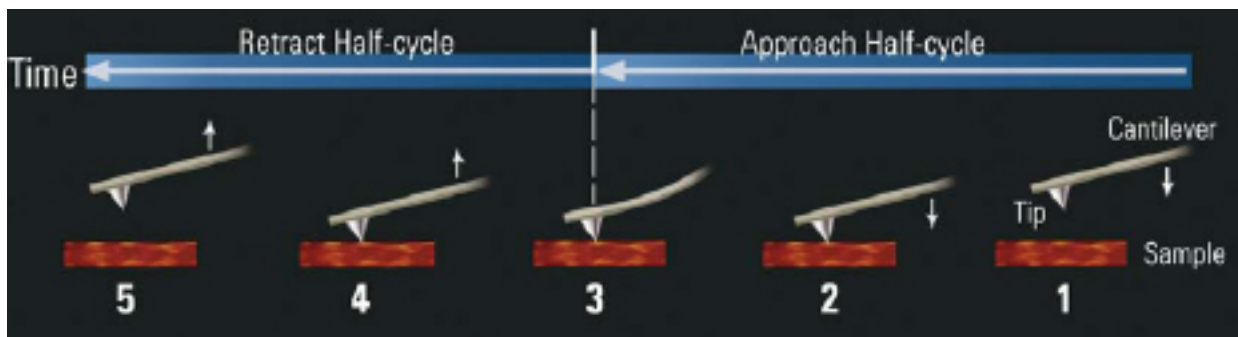


Figure 107 Schematic overview of force spectroscopy

Force spectroscopy is widely used in air, liquids, and different controlled environments. It may involve functionalized tips to study specific interactions of conjugated molecules or chemically-modified surfaces.

In order to quantitatively measure interaction forces, it is necessary to know the bending stiffness, also referred to as the spring or force constant, of the cantilever as accurately as possible. This is described in more detail in "[Thermal K Calibration](#)" on page 281.

The procedure described below covers general guidelines for Force Spectroscopy in Contact Mode. It is a user-dependent choice to select Contact vs AC Mode. The choice of scanning prior to taking force measurements also is user-dependent.

If AC Mode is chosen, note that the **Contact Mode Sweep** check box in the Spectroscopy window Advanced tab may be used so the imaging

mode does not have to be changed to acquire Force vs Distance measurements.

If scanning is done prior to taking force measurements and a specific location is of interest, use the Point and Click tool described in the online PicoView User Guide to **Move** the tip to that position.

Force Spectroscopy Procedure for Contact Mode

This procedure is done after the system has been prepared for Contact Mode imaging as described in [Chapter 4](#), "Preparing for Imaging" and [Chapter 5](#), "Contact Mode Imaging".

For more accurate measurements, use a Closed Loop scanner and follow the CL scanner calibration prior to acquiring Force vs. Distance data. Select the **CL Sweep** check box in the Setup tab of the Spectroscopy window.

- 1 Choose **Mode > Contact**.
- 2 In the Servo dialog box ([Figure 108](#)), set the following parameters as shown below:
 - a Set Gains as needed for the sample.
 - b Set the Setpoint to **0 V**.
 - c Maximize the Range by entering a very large number (ie. 999) and PicoView will adjust to the maximum value for the scanner.

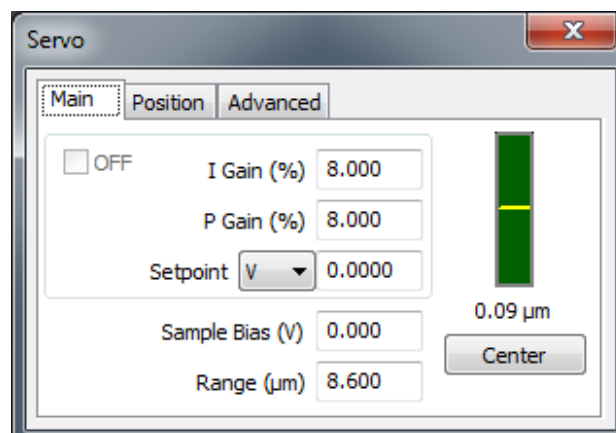




Figure 108 Initial Servo settings for Force Spectroscopy

3 Choose **Controls > Spectroscopy** or click the **Spectroscopy** toolbar button  to open the Spectroscopy window.

4 Verify the Plot Type is set to **Force vs Distance**, either in the Spectroscopy drop down menu or in the status bar at the bottom of the Spectroscopy window.

5 **Approach** the tip.

6 In the Spectroscopy Basic tab, set the Sweeps as needed to either 1 or .

7 In the Spectroscopy Setup tab, set the Duration to **1**.

8 Set the Data Points to **200**.

9 Maximize the Start and minimize the End values:

- Enter a large number for the Start value (ie 999) and PicoView will adjust to the maximum value for the scanner.

- Enter a large negative number for the End value (ie. -999) and PicoView will adjust to the minimum value for the scanner.

10 If using a calibrated CL scanner, select the **CL Sweep** check box (as shown in [Figure 109](#)) in the Z Closed Loop section of the Setup tab.

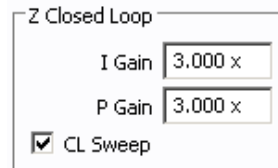
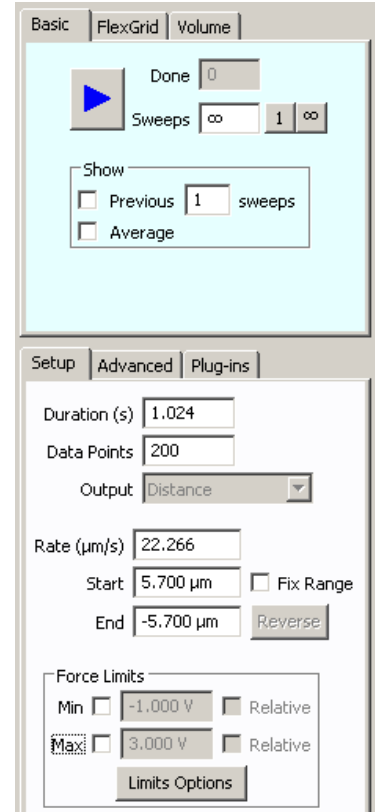


Figure 109 Z closed loop section of Spectroscopy Setup tab with **CL Sweep** check box selected

To save successive force measurements, have files saved automatically after each force curve:

- 1 In the Spectroscopy window, choose **File > Set filename ...**.
- 2 In the **Save As** dialog box, enter a filename for the data set. Note the file type will default to *.mi.
- 3 Click the **Save** button.

Turn on Auto Save by choosing **File > Auto Save**. The title bar at the top of the window will indicate Auto Save is on. It will also be indicated by a check mark next to Auto Save in the Spectroscopy File menu.

Begin to take force measurements. For the initial acquisition, it is recommended that Toggle Auto Full Scale be turned **On**.

- 1 Start the sweep by clicking the **Start** button.
- 2 Stop measurements at any time by clicking the **Stop** button.
- 3 After the initial sweep has completed, adjust the sweep range as needed using the Start/End parameters in the Spectroscopy Setup tab.

An example of a closed loop Force vs Distance sweep is shown in Figure 110.

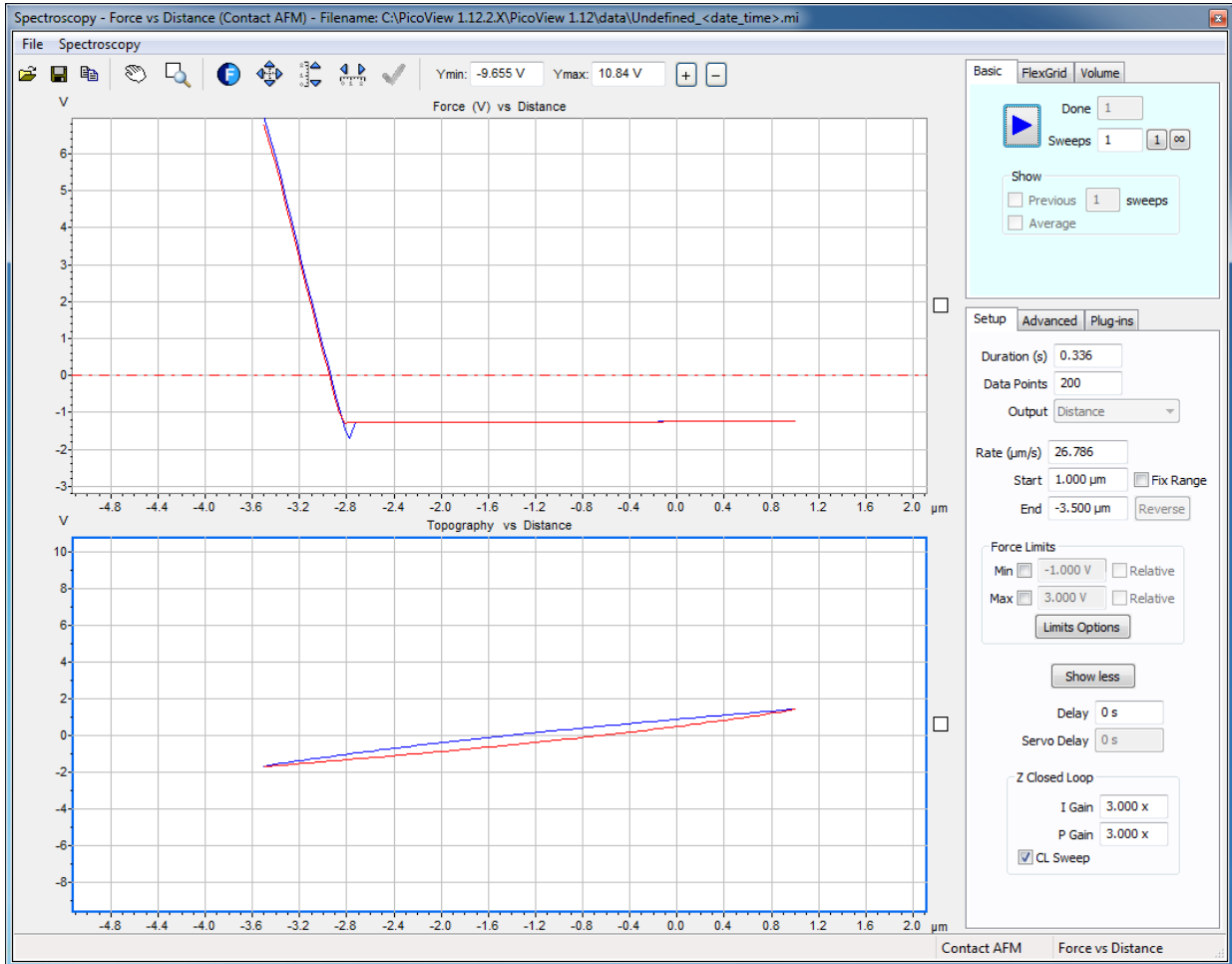


Figure 110 Closed loop force spectroscopy example

FlexGrid Spectroscopy

FlexGrid Spectroscopy is an extension of Force spectroscopy that is used to measure the polarity and strength of the interaction between the AFM tip and the sample at user defined points. Math plug-ins are used to extract material properties such as adhesion and stiffness. In FlexGrid Spectroscopy, a standard topography image is collected and the user selects specific areas of the image to analyze.

The procedure for FlexGrid Spectroscopy is similar to Force Spectroscopy, with the additional steps of collecting a topographic image before performing spectroscopic measurements and using math plug-ins to extract the desired material properties after collecting spectroscopic data.

The procedure described below covers general guidelines for FlexGrid Spectroscopy in Contact Mode. It is a user-dependent choice to select Contact vs AC Mode.

FlexGrid Spectroscopy Procedure for Contact Mode

This procedure is done after the system has been prepared for Contact Mode imaging as described in [Chapter 4](#), "Preparing for Imaging".

For more accurate measurements, use a Closed Loop scanner and follow the CL scanner calibration prior to acquiring Force vs. Distance data. Select the **CL Sweep** check box in the Setup tab of the Spectroscopy window.

- 1 Collect a topographic image as described in [Chapter 5](#), "Contact Mode Imaging".
- 2 Choose **Mode > Contact**.
- 3 In the Servo dialog box ([Figure 111](#)), set the following parameters as shown below:
 - a Set Gains as needed for the sample.
 - b Set the Setpoint to **0 V**.
 - c Maximize the Range by entering a very large number (ie. 999) and PicoView will adjust to the maximum value for the scanner.

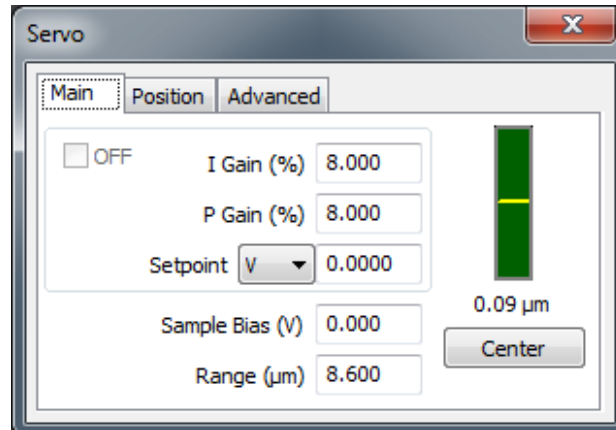




Figure 111 Initial Servo settings for FlexGrid Spectroscopy

4 Choose **Controls >**

Spectroscopy or click the **Spectroscopy** toolbar button  to open the Spectroscopy window.

5 Verify the Plot Type is set to **Force vs Distance**, either in the Spectroscopy drop down menu or in the status bar at the bottom of the Spectroscopy window.

6 **Approach** the tip.

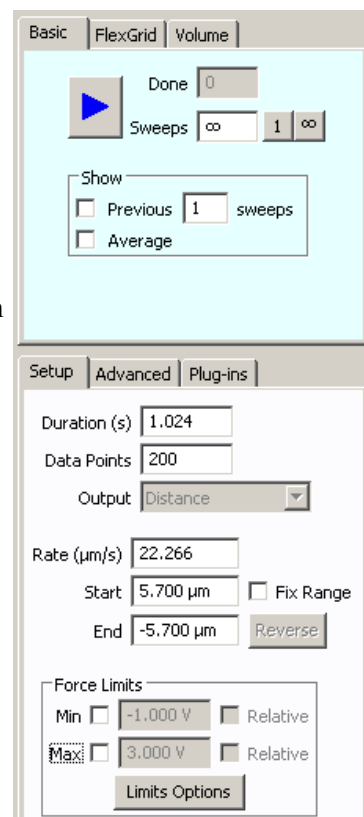
7 In the Spectroscopy Basic tab, set the Sweeps as needed to either **1** or .

8 In the Spectroscopy Setup tab, set the Duration to **1**.

9 Set the Data Points to **200**.

10 Maximize the Start and minimize the End values

- a Enter a large number for the Start value (ie 999) and PicoView will adjust to the maximum value for the scanner.
- b Enter a large negative number for the End value (ie. -999) and PicoView will adjust to the minimum value for the scanner.



- 11 If using a calibrated CL scanner, select the **CL Sweep** check box (as shown in [Figure 112](#)) in the Z Closed Loop section of the Setup tab.

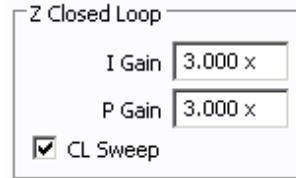


Figure 112 Z closed loop section of Spectroscopy Setup tab with **CL Sweep** check box selected

- 12 Click the **FlexGrid** tab in the Spectroscopy window to display the topographic image. The transparency of the image can be adjusted by clicking on it and rolling the mouse wheel.
- 13 Using the **Add Point** tool in the Spectroscopy window, click on one or more areas of interest in the image (as shown in [Figure 113](#)).

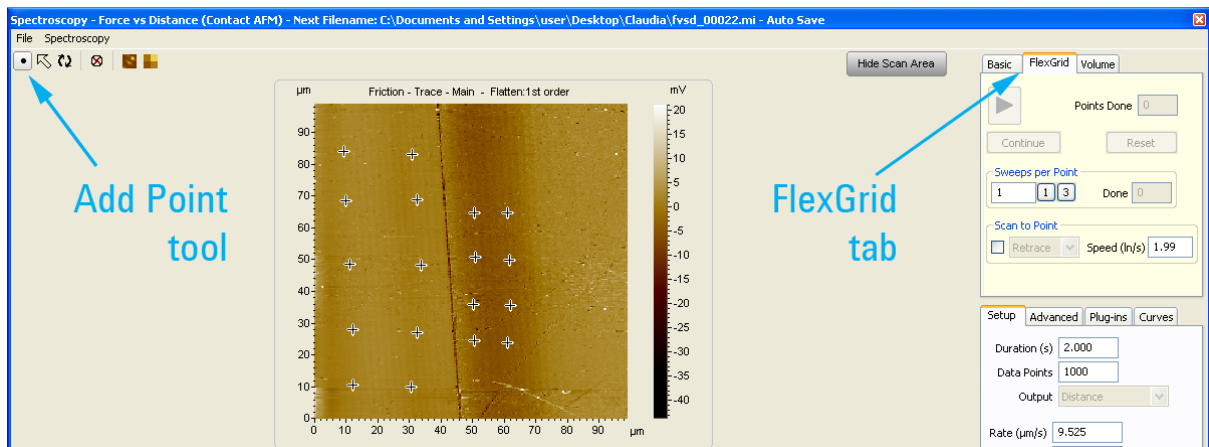


Figure 113 FlexGrid tab of the Spectroscopy window with specific points in the topographic image selected using the Add Point tool.

To save successive force measurements, have files saved automatically after each force curve:

- 1 In the Spectroscopy window, choose **File > Set filename ...**.
- 2 In the **Save As** dialog box, enter a filename for the data set. Note the file type will default to *.mi.
- 3 Click the **Save** button. Turn on Auto Save by choosing **File > Auto Save**. The title bar at the top of the window will indicate Auto Save is on. It will also be indicated by a check mark next to Auto Save in the Spectroscopy File menu.

Begin to take force measurements. For the initial acquisition, it is recommended that Toggle Auto Full Scale be turned **On**.

- 1 Start the sweep of selected points by clicking the **Start** button.
- 2 Stop measurements at any time by clicking the **Stop** button.
- 3 After the initial sweep has completed, adjust the sweep range as needed using the Start/End parameters in the Spectroscopy Setup tab.

NOTE

The scan to point section of the FlexGrid tab allows image and spectroscopy data to be collected simultaneously.

After force measurements have been collected, extract the material properties and generate plug-in maps.

- 1 In the Spectroscopy window, click the **Show Plug-in Images** tool.
- 2 In the Plug-in Images window, click the “+” button to add to add up to eight windows (Figure 114).
- 3 To set what each window will display, select the desired spectroscopy and math plug-in inputs from the pull down menus. The lists will vary depending on the imaging mode.

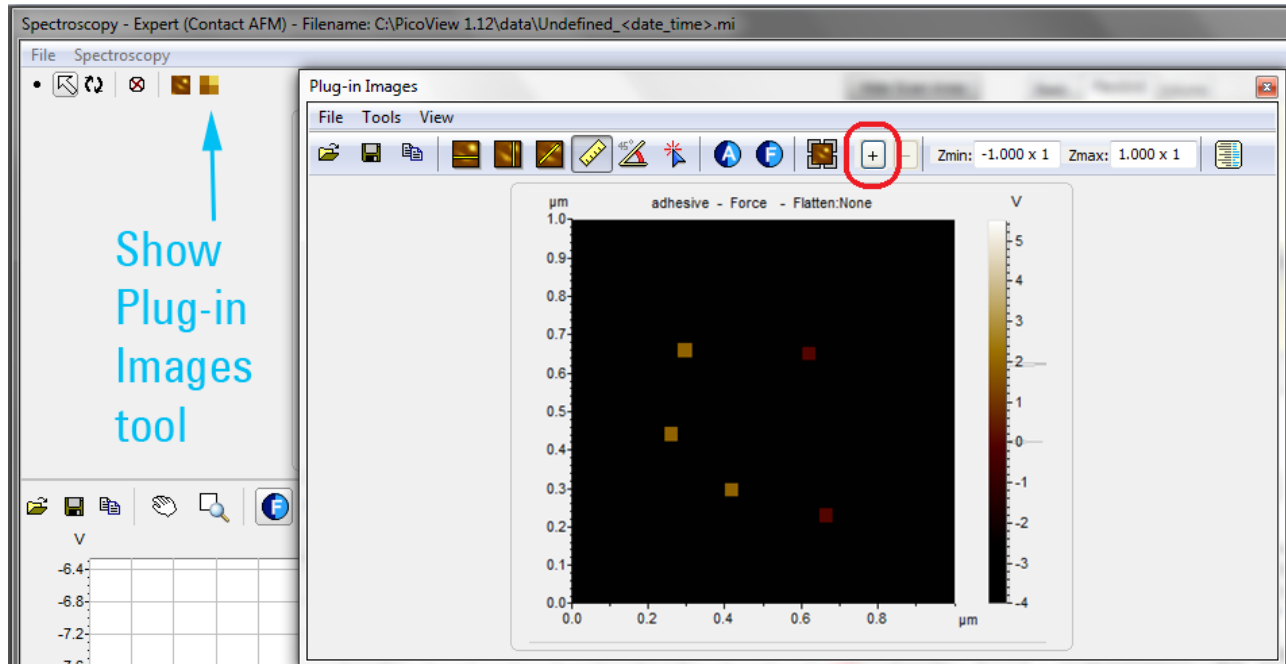


Figure 114 Plug-in Images window

Volume Spectroscopy

Volume Spectroscopy is also an extension of Force Spectroscopy. The polarity and strength of the interaction between the AFM tip and the sample is measured in user defined areas. Math plug-ins are used to extract parameters such as stiffness and adhesion to produce a two dimensional map of material properties. As in FlexGrid Spectroscopy, a standard topography image is collected and the user selects a particular volume of the image to analyze.

The procedure for Volume Spectroscopy is the same as for FlexGrid Spectroscopy, but the user defines a volume to analyze, rather than individual points. The resolution of the measurements is defined by the volume of analysis, with up to image pixel resolution points. The procedure described below covers general guidelines for Volume Spectroscopy in Contact Mode. It is a user-dependent choice to select Contact vs AC Mode.

Volume Spectroscopy Procedure for Contact Mode

This procedure is done after the system has been prepared for Contact Mode imaging as described in [Chapter 4](#), "Preparing for Imaging".

For more accurate measurements, use a Closed Loop scanner and follow the CL scanner calibration prior to acquiring Force vs. Distance data. Select the **CL Sweep** check box in the Setup tab of the Spectroscopy window.

- 1 Collect a topographic image as described in [Chapter 5](#), "Contact Mode Imaging".
- 2 Choose **Mode > Contact**.
- 3 In the Servo dialog box ([Figure 115](#)), set the following parameters as shown below:
 - a Set Gains as needed for the sample.
 - b Set the Setpoint to **0 V**.
 - c Maximize the Range by entering a very large number (ie. 999) and PicoView will adjust to the maximum value for the scanner.

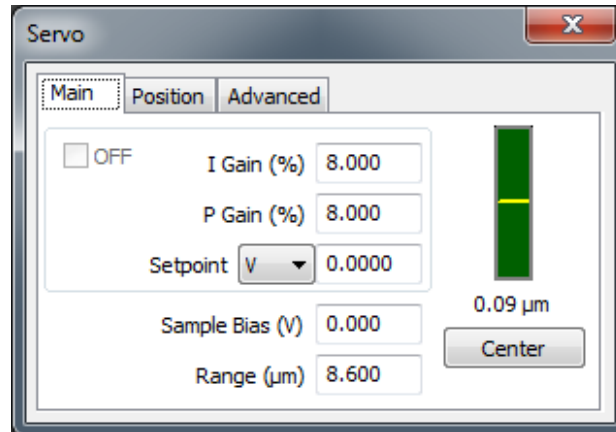


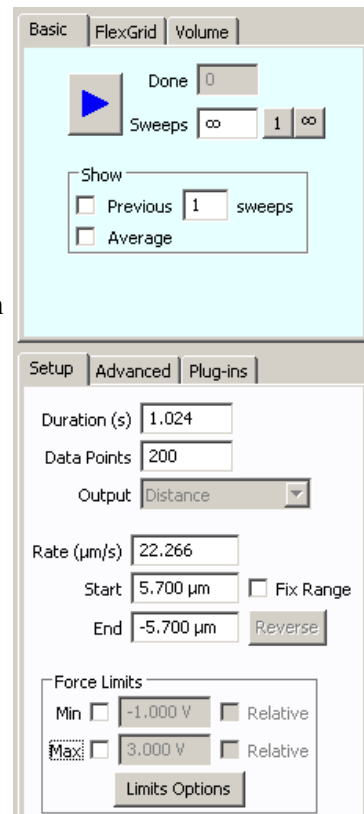


Figure 115 Initial Servo settings for Volume Spectroscopy

- 4 Choose **Controls** > **Spectroscopy** or click the **Spectroscopy** toolbar button  to open the Spectroscopy window.
- 5 Verify the Plot Type is set to **Force vs Distance**, either in the Spectroscopy drop down menu or in the status bar at the bottom of the Spectroscopy window.
- 6 **Approach** the tip.
- 7 In the Spectroscopy Basic tab, set the Sweeps as needed to either 1 or .
- 8 In the Spectroscopy Setup tab, set the Duration to 1.
- 9 Set the Data Points to 200.
- 10 Maximize the Start and minimize the End values
 - a Enter a large number for the Start value (ie 999) and PicoView will adjust to the maximum value for the scanner.
 - b Enter a large negative number for the End value (ie. -999) and PicoView will adjust to the minimum value for the scanner.



- 11 If using a calibrated CL scanner, select the **CL Sweep** check box (as shown in **Figure 116**) in the Z Closed Loop section of the Setup tab.

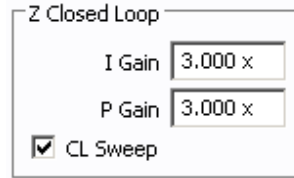


Figure 116 Z closed loop section of Spectroscopy Setup tab with **CL Sweep** check box selected

- 12 Click the **Volume** tab in the Spectroscopy window to display the topographic image. The transparency of the image can be adjusted by clicking on it and rolling the mouse wheel.
- 13 Specify the resolution by selecting a volume from the pull down menu.
- 14 Using your mouse, click in the topographic image and drag a rectangle to specify which force curves will be displayed in the spectroscopy window.

To save successive force measurements, have files saved automatically after each force curve:

- 1 In the Spectroscopy window, choose **File > Set filename ...**.
- 2 In the **Save As** dialog box, enter a filename for the data set. Note the file type will default to *.mi.
- 3 Click the **Save** button. Turn on Auto Save by choosing **File > Auto Save**. The title bar at the top of the window will indicate Auto Save is on. It will also be indicated by a check mark next to Auto Save in the Spectroscopy File menu.

Begin to take force measurements. For the initial acquisition, it is recommended that Toggle Auto Full Scale be turned **On**.

- 1 Start the sweep of the selected area by clicking the **Scan Up or Scan Down** buttons.
- 2 Stop measurements at any time by clicking the **Stop** button.
- 3 After the initial sweep has completed, adjust the sweep range as needed using the Start/End parameters in the Spectroscopy Setup tab.

An example of a Volume Spectroscopy sweep is shown in Figure 117.

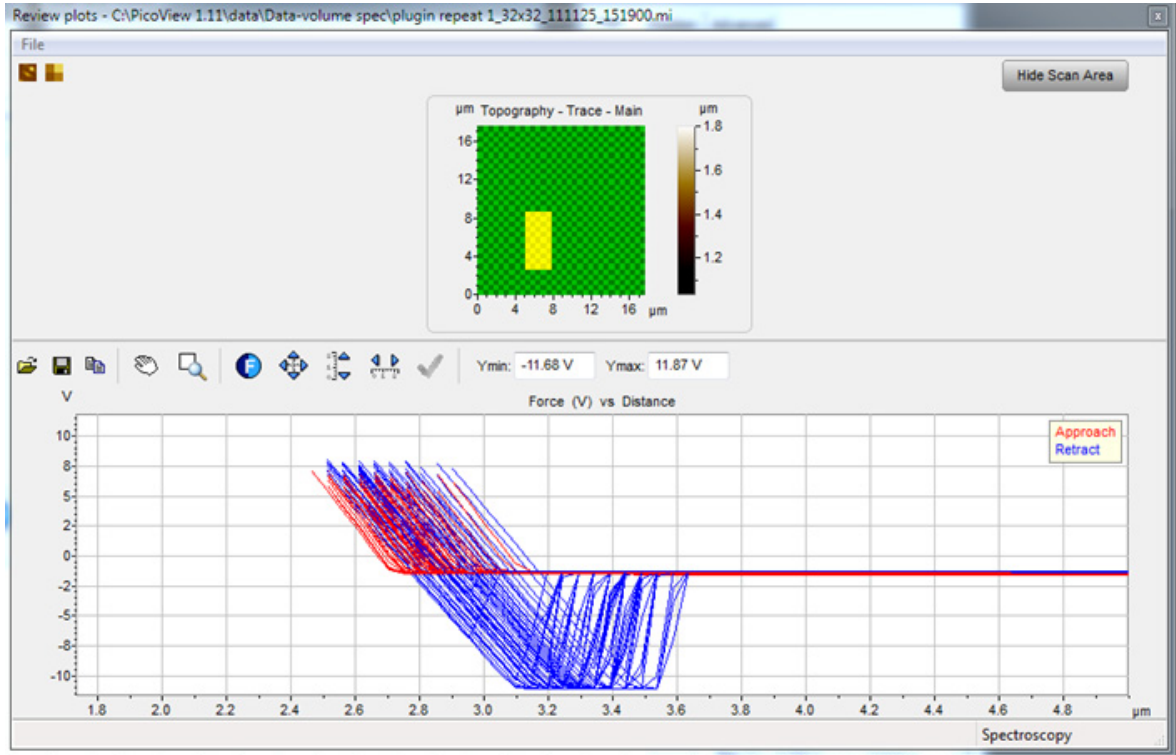


Figure 117 Volume spectroscopy sweep example

NOTE

As for FlexGrid Spectroscopy, the scan to point section of the Volume tab allows image and spectroscopy data to be collected simultaneously.

After force measurements have been collected, extract the material properties and generate plug-in maps.

- 1 In the Spectroscopy window, click the **Show Plug-in Images** tool.
- 2 In the Plug-in Images window, click the “+” button to add up to eight windows.
- 3 To set what each window will display, select the desired spectroscopy and math plug-in inputs from the pull down menus. The lists will vary depending on the imaging mode.

Several examples of volume spectroscopy plug-in windows with different inputs selected are shown in [Figure 118](#).

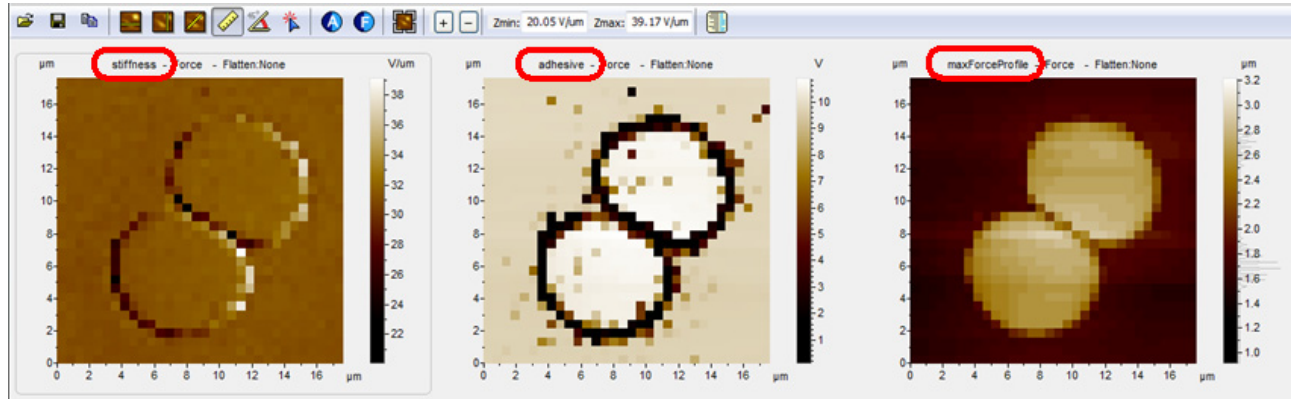


Figure 118 Volume spectroscopy plug-in windows with inputs selected

The results of volume spectroscopy plug-in analysis and two dimensional maps of material properties are shown in [Figure 119](#).

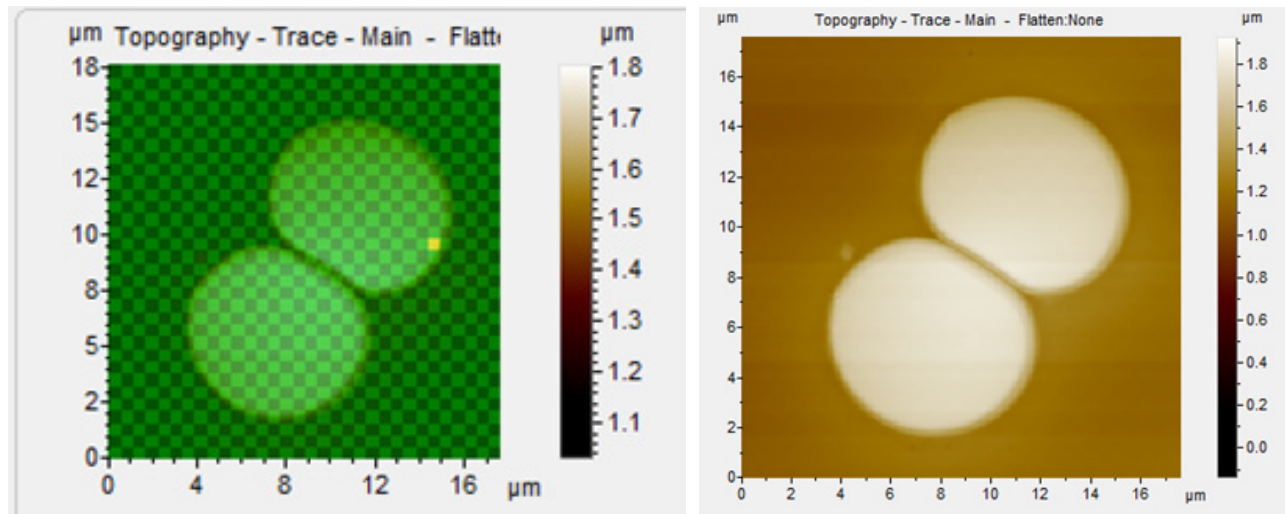


Figure 119 Output of volume spectroscopy plug-in window analysis

8 Scanner Maintenance and Calibration

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Keysight scanners are designed for years of consistent, worry-free operation. However, scanners contain extremely delicate components and must be treated with care to maintain their high level of operation. This chapter discusses regular maintenance of the scanner, nose assemblies and probes, as well as regular calibration procedures for the multi-purpose, open-loop scanner.

Care and Handling of the Probes and Scanner

Probes

Always store probes at room temperature in their protective cases.

Handle gently with tweezers, following only the described procedures.

If a probe is dropped it may very well be damaged. You can check whether the cantilever is intact by viewing it through a loupe or other magnifying device.

If you are using more than one type of probe, be sure to store them separately in well-marked cases to avoid confusion.

Nose Assembly

Store nose assemblies in a clean, dry location where they will not be subject to excessive humidity, temperature changes or contact.

The scanner fixture is designed with a socket to safely hold a nose assembly while you are working with the scanner. This is not a permanent storage location, but it is a safe way to keep the nose assembly close at hand.

Dirt, grease or spots on the glass window of the nose assembly can interfere with the optical path of the laser. Regularly remove the probe and clean the nose assembly window with cotton or a soft tissue (dry, wetted with water, or with ethanol).

The glass is glued to the nose assembly with chemically resistive epoxy, so if the window breaks there is no easy way to replace it and the entire

nose assembly will likely need to be replaced. Use careful handling to avoid damaging the window.

Only remove the nose assembly from the scanner using the Nose Assembly Removal Tool, with the scanner placed upright in its fixture.

Do NOT use the Removal Tool to install the nose assembly in the scanner.

Two-Piece Nose Cone Cleaning

The two-piece nose cone is not to be used in liquid because it does not have a glass window to prevent liquid from getting to the scanner. After it is removed from a scanner, the two-piece nose cone may be cleaned with a low oxidizing organic solvent such as ethyl alcohol.

Scanner

Between uses, remove the scanner from the microscope and store it, on its assembly fixture or in its storage case, in a location where it will not be subject to excessive humidity, temperature changes or contact. Keysight recommends that scanners be stored in a desiccator.

Use care when moving the scanner on its assembly stand as it is not secured to the stand and can be damaged if dropped.

The scanner contains very brittle and fragile piezoelectric ceramic components. Applying excessive lateral force while exchanging nose assemblies, or dropping the scanner even a short distance onto a hard surface, will permanently damage the scanner. Use only the procedures described in [Chapter 4](#) to install and remove the nose assembly.

If the nose assembly housing becomes loose or can be wiggled with the fingers when in place, contact Keysight support for assistance.

Cracked or broken piezo components will result in abnormal imaging. Damage to the scanner such as those described above are NOT covered by the standard warranty.

Scanner Characteristics

The multi-purpose scanner includes several piezoelectric elements for moving the probe along the X, Y and Z axes. Piezoelectric materials inherently exhibit non-ideal properties, the effects of which are

explained below. Though they are explained separately for simplicity, they may not be independent of each other.

All Keysight scanners are calibrated before shipment and installation. A unique calibration file is provided with each system, as is a “generic” calibration file, which is not scanner-specific.

Non-Linearity

Figure 120 shows a calibration target consisting of square features of known size and equal spacing. The image was made with an uncalibrated scanner. Features are larger at the start of each scan line, and also at the bottom of the image. This effect is the result of non-linearity of the piezo response across the scan range.

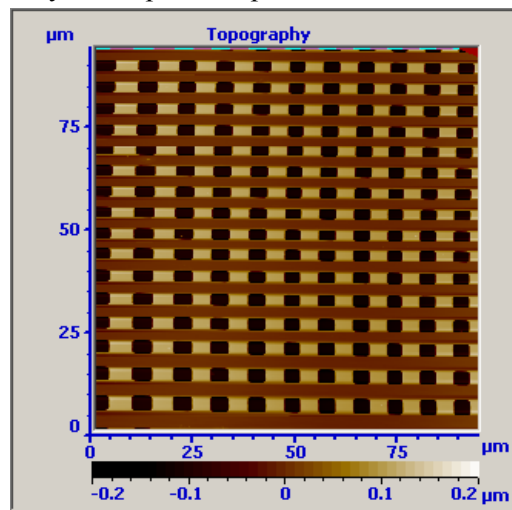


Figure 120 Image from an uncalibrated scanner showing non-linearity

Sensitivity

The features in Figure 120 also change size within each scan line. Lateral feature sizes may be reported incorrectly due to changes in piezo sensitivity.

Hysteresis

Hysteresis is an effect in which the piezo movement during extension in one direction does not match the movement during contraction caused by an equal and opposite field in the other direction. The effect of

hysteresis is that the trace will be offset from the retrace, as in Figure 121.

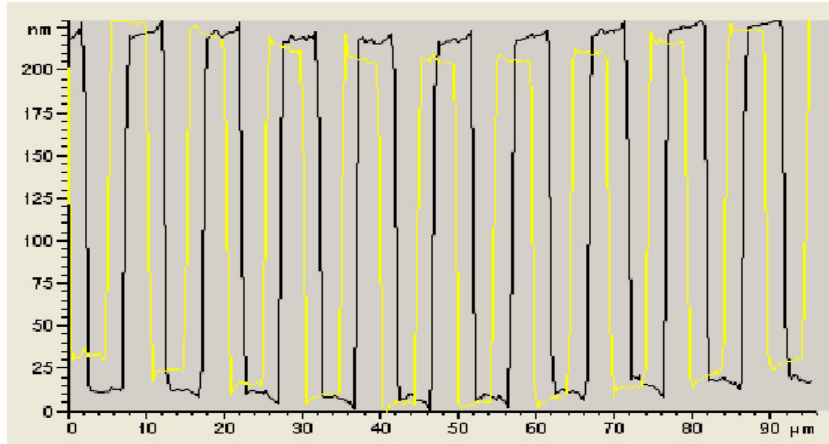


Figure 121 Scanner hysteresis before correction. The yellow line is the Trace image, and the blue is the Retrace.

Other Characteristics

Bow

During raster scanning, the free end of the scanner moves in an arc over the full range of the scanner, as opposed to a flat line in a plane above the sample. Bowing is minimized by the “balanced pendulum” design of the Keysight scanners. Residual bowing is typically accounted for by “flattening” algorithms in the PicoView software.

Cross Coupling

Cross coupling is the effect in which movement of the scanner along one axis (usually X or Y) causes unwanted movement along the other axis (Z). Systems using tube scanners are more susceptible to geometric cross coupling because a single four-quadrant tube provides motion in all the three axes. The larger Keysight multi-purpose scanners, with 90 micron X/Y scan range, use separate piezoelectric elements for X/Y movement and for Z movement. This configuration helps to reduce the cross coupling between different axes. Smaller range scanners (e.g., 10 micron X/Y scan range) use two sets of plates (one each for X and Y

movement) and a single tube for Z motion. Since the range is small with these scanners, the effect of cross-coupling is minimal.

Aging

Aging is a time-dependent effect in which the sensitivity (extension per unit of field) of the scanner decreases, approximately exponentially, over time. A large amount of decrease takes place during the first few hours of use. Therefore, scanners are burned in before initial calibration. After this initial burn-in, the aging rate is very slow; however, calibration once or twice per year is still recommended.

Creep

At a constant applied voltage the piezo position will change slightly over time, most noticeably at the beginning of a scan or after a change on scan location. Creep appears as elongation of the feature in the direction of the offset because of this slow movement (Figure 122).

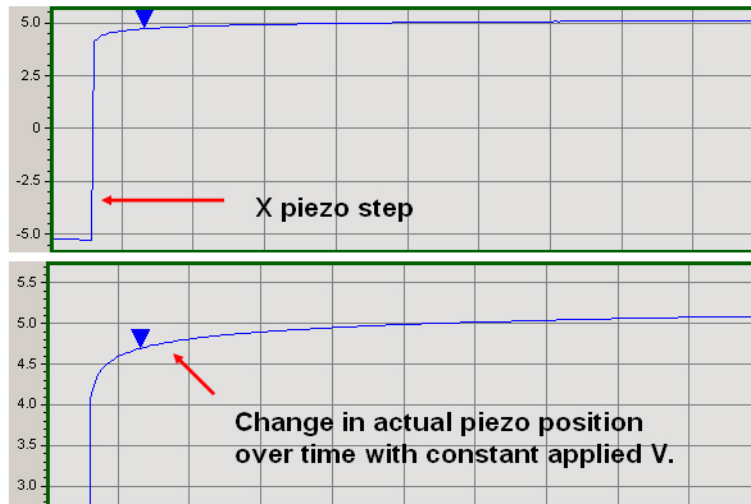


Figure 122 Scanner creep

Calibrating the Multi-Purpose Scanner

Regular calibration ensures that Keysight multi-purpose scanners will provide high performance imaging for many years of service. The following calibration procedure is recommended once or twice per year, if the system is moved, and before critical measurements.

NOTE

The procedures and descriptions in this User Guide are for PicoView 1.12x and higher.

- 1 Make sure the correct scanner file is selected under the PicoView **Scanner** menu.
- 2 Place a calibration target on a sample plate and mount the plate on the microscope.
- 3 Follow the procedures for Contact Mode measurements to obtain an image of the calibration target. Use the following settings:
 - a Deflection = **-0.8 to -1.0 V**.
 - b Setpoint voltage = **0 V**.
 - c Scan Size = **90 microns**.
 - d Resolution = **256**.
 - e Scan Speed to **1 ln/s**.
- 4 Make sure that the target is positioned such that its features are aligned in both the X and Y directions. Use a **Cross Section** tool (Horizontal, Vertical or Real Time) in the Realtime Images window, from either the Tools Menu or Toolbar, as an aid. If the target is not aligned, withdraw the tip, adjust the target, and approach again.
- 5 In the Realtime Images window, choose **File > Autosave**. During calibration this provides a useful way to review the effects of applied changes to the calibration file.

X Calibration

In the Realtime Images window, set up two Topography images, one for **Trace** and one for **Retrace** (Figure 123).

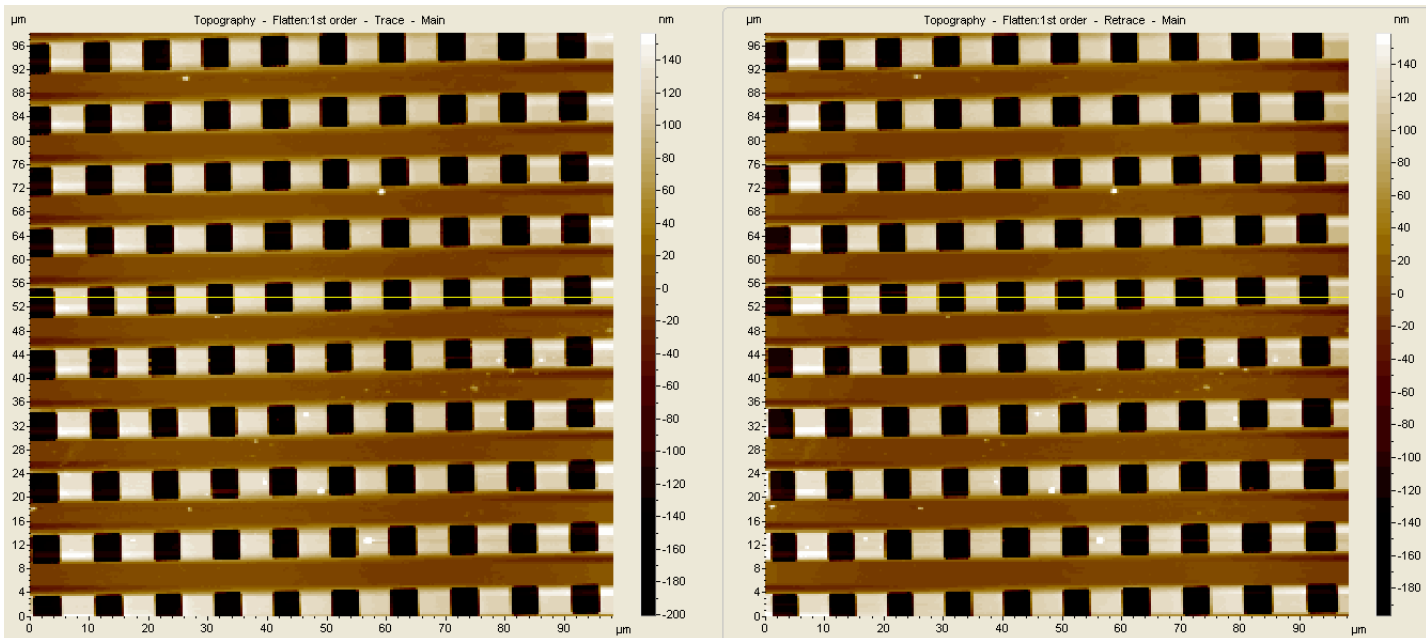


Figure 123 Images of calibration target during Trace and Retrace

- 6 Choose **Scanner > Edit** to open the Scanner Setup dialog box. Select the Open Loop tab.

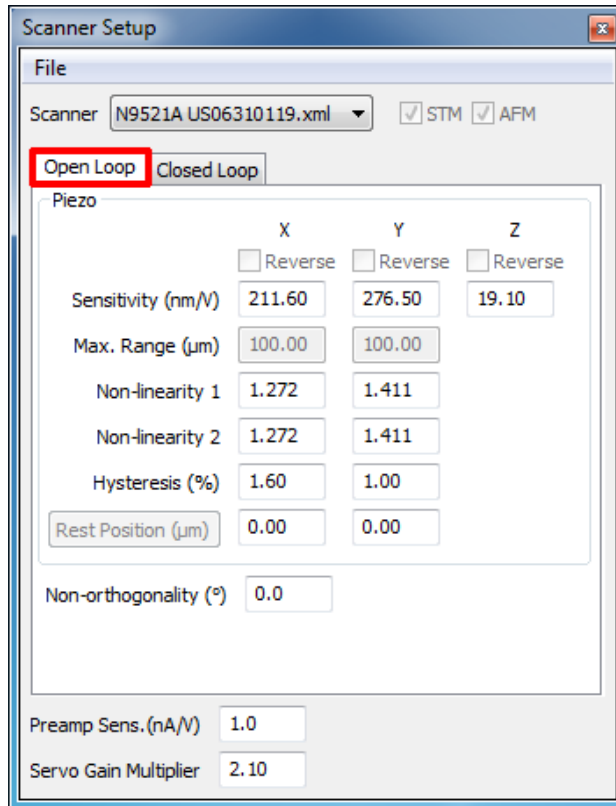


Figure 124 Scanner Setup dialog box

X Non-Linearity

In the Realtime Images window, choose **Tools > Horizontal Cross Section**. Use markers to report the dimensions between sets of features at either end of the scan range ([Figure 125](#)).

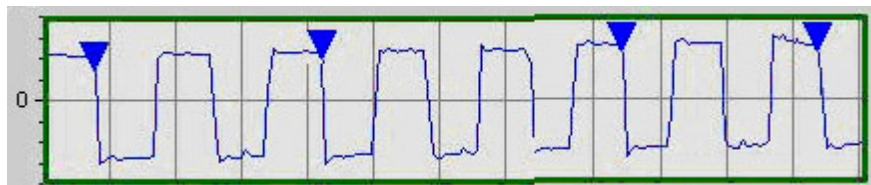


Figure 125 Horizontal cross-section tool used to check non-linearity. Two sets of cursors are shown.

If the spacing is not identical for the two sets of features, adjust the X Non-linearity terms in the Open Loop tab of the Scanner Setup dialog box, according to the following equation:

$$NewTerm = \frac{StartSize \times CurrentTerm}{EndSize}$$

where

StartSize = size of features at the start of the scan

EndSize = size of features at the end of the scan

CurrentTerm = current non-linear correction term.

Adjust **Non-linearity 1** for the Trace scan and adjust **Non-linearity 2** using the Retrace scan.

You can also use the diagram in [Figure 126](#) as a guide.

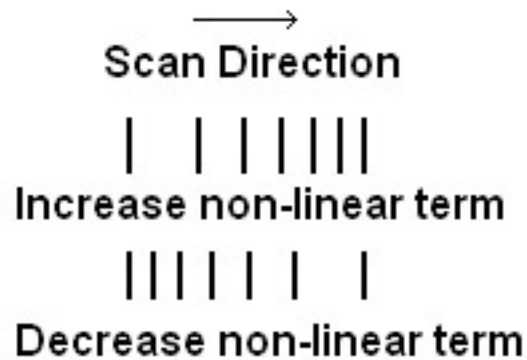


Figure 126 Correction diagram for X non-linearity

After adjusting the X Non-linearity parameters, re-scan the calibration target. The spacing between features should be approximately the same across the scan range for the Trace and Retrace scans.

X Hysteresis

Place a vertical cursor at the same location in the Trace and Retrace images. The cursor should cross the same features in both images. If this

is not the case, as in Figure 127, increase the X Hysteresis value and re-scan.

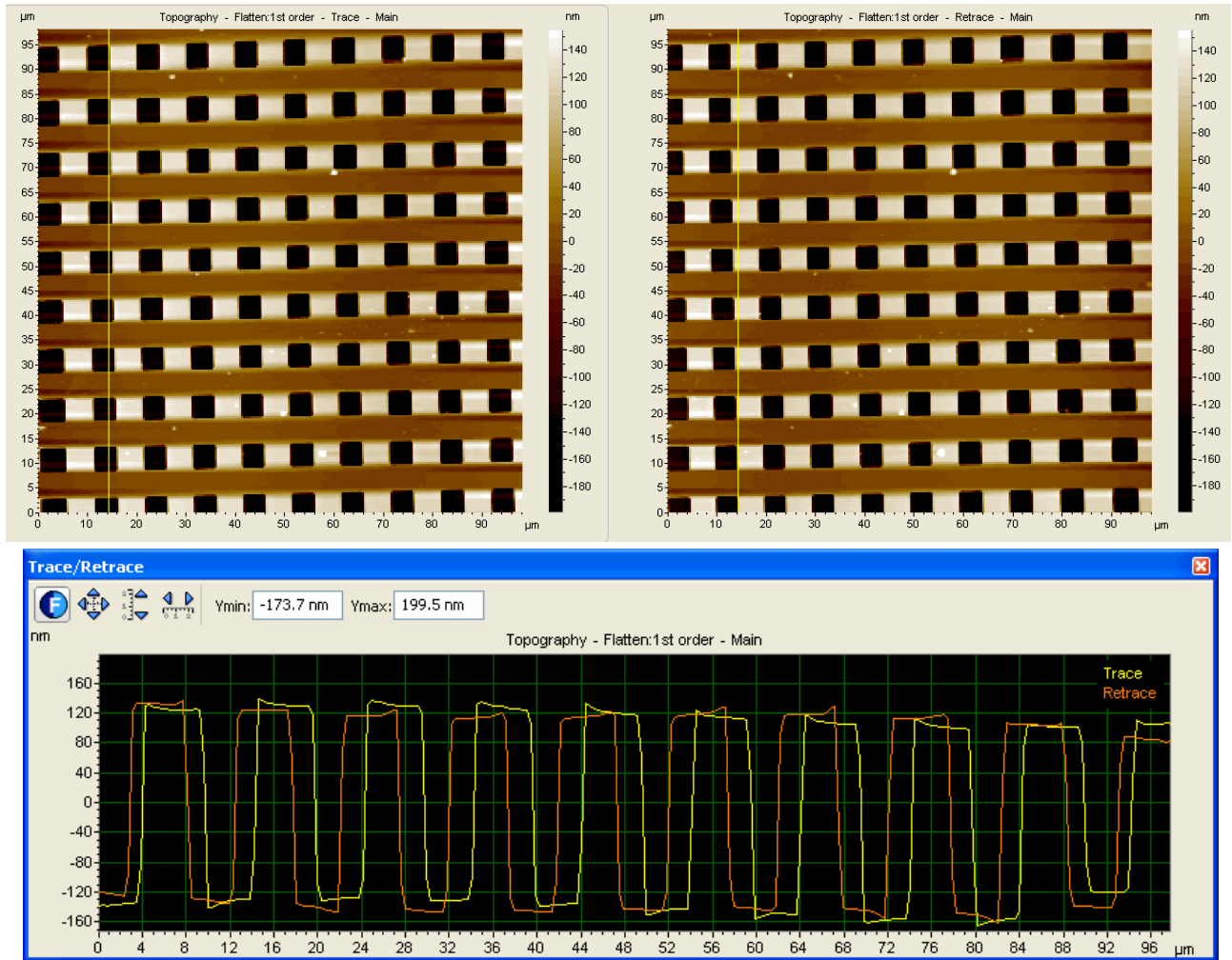


Figure 127 Misalignment between trace and retrace

Alignment should be consistent across the full range of the X axis, as shown below in **Figure 128**. Several iterations may be necessary to align all edges in the Trace and Retrace images.

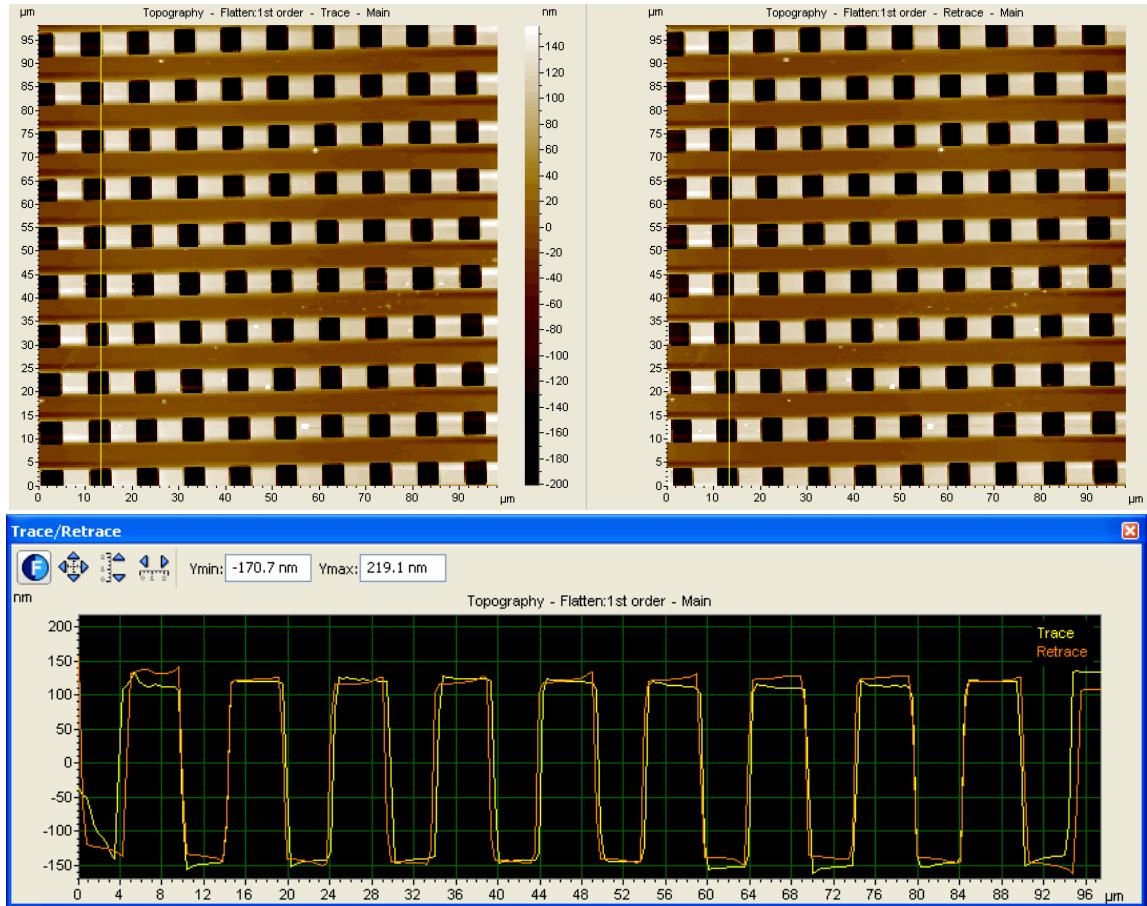


Figure 128 Features align properly after X Hysteresis adjustment

X Sensitivity

Using the Cross Section tool from the Realtime Images window, measure the length of a set of features across the scan (**Figure 129**).



Figure 129 Cross-section of several features to check X Sensitivity

If the measured dimension does not match the actual, then adjust the X Sensitivity term according to the following equation:

$$NewSensitivity = \frac{CurrentSensitivity \times KnownSize}{MeasuredSize}$$

After adjustment the measured size should be within 5 % of the actual size.

Y Calibration

The methods used for calibrating the scanner's Y axis are similar to those used for the X dimension. The exception is that the scan range will be set to ½ of the scanner's full range. The Y axis is generally used as the slow scanning axis so the range will be reduced as a time consideration.

Only one Topography image is required for Y calibration. The other image can be assigned to display flattened Topography data.

Y Non-Linearity

Obtain an image of the calibration target. Using the Cross Section tool, set markers at the uppermost and bottommost feature along a vertical cross section (Figure 130).



Figure 130 Cross-section in Y direction showing non-linearity

If the dimensions are not identical for the two features, adjust the Y Non-linearity terms according to the equation:

$$NewTerm = \frac{StartSize \times CurrentTerm}{EndSize}$$

where

StartSize = size of features at the start of the scan

EndSize = size of features at the end of the scan

CurrentTerm = current non-linear correction term.

Adjust **Non-linearity 1** for the Up scan and adjust **Non-linearity 2** using the Down scan.

Use the diagram in **Figure 131** as a guide for making the correction.

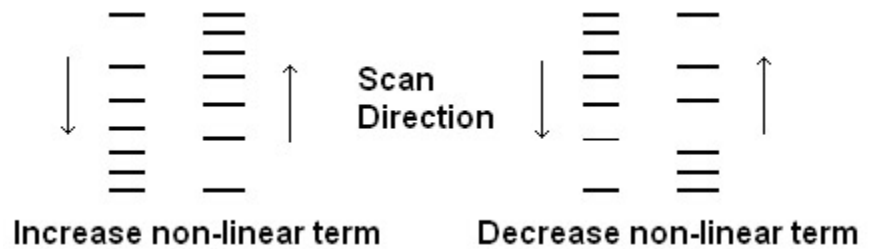


Figure 131 Correction diagram for Y Non-linearity

After adjusting the Y Non-linearity parameters, re-scan the calibration target. The spacing between features should be approximately the same across the scan range for the Trace and Retrace scans.

Y Hysteresis

The next step will be to adjust the Y Hysteresis term. Assign one data channel to display a single frame of the calibration target and place a vertical cross-section through a line of features. Allow the scanner to scan continuously, which will update the Cross Section plot each pass through the frame.

Figure 132 shows the upward scan (blue marker) and downward re-trace (red marker) data. The markers are used to measure the difference in the acquired data at a given point on the Y axis. The blue marker was placed on the edge of a feature while the scan was moving upwards. The red marker was placed on the same feature during the downward scan.

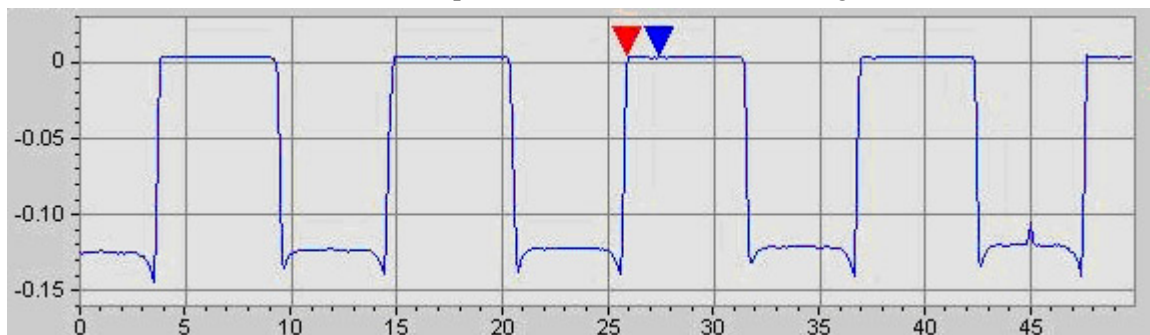


Figure 132 Markers indicating trace and re-trace Y hysteresis

While scanning in one direction, focus on one step of the grating. As the scan data is plotted, position a marker on this edge. Wait for the scan in the opposite direction to occur and position a second marker on the same edge after the plot has been updated. In the example below, about 2 microns of hysteresis can be measured using this method.

After increasing or decreasing the Y Hysteresis term, alignment of the individual edges should be confirmed across the full range of the Y axis.

It may be necessary to update the hysteresis term more than once before all edges become aligned in both the upward and downward scans.

Y Sensitivity

Using the Vertical Cross Section tool in the Realtime Images window (from the Tools menu or Toolbar), measure the top-to-bottom distance of a set of features across the scan (Figure 133).

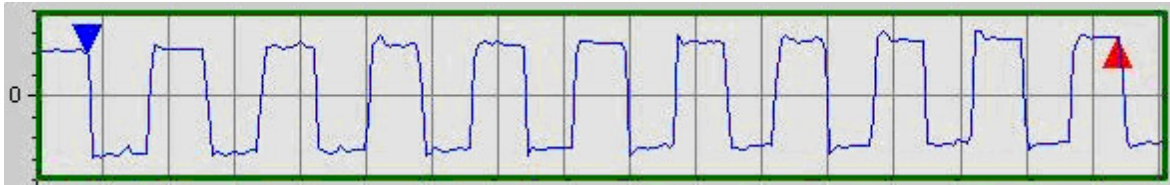


Figure 133 Cross-section of several features to check Y sensitivity

If the measured dimension does not match the actual, the adjust the Y Sensitivity term according to the following equation:

$$NewSensitivity = \frac{CurrentSensitivity \times KnownSize}{MeasuredSize}$$

Non-Orthogonality

Using the Angle tool, measure the angle between features at all four corners of the image. The image below (Figure 134) shows one of the four corner measurements.

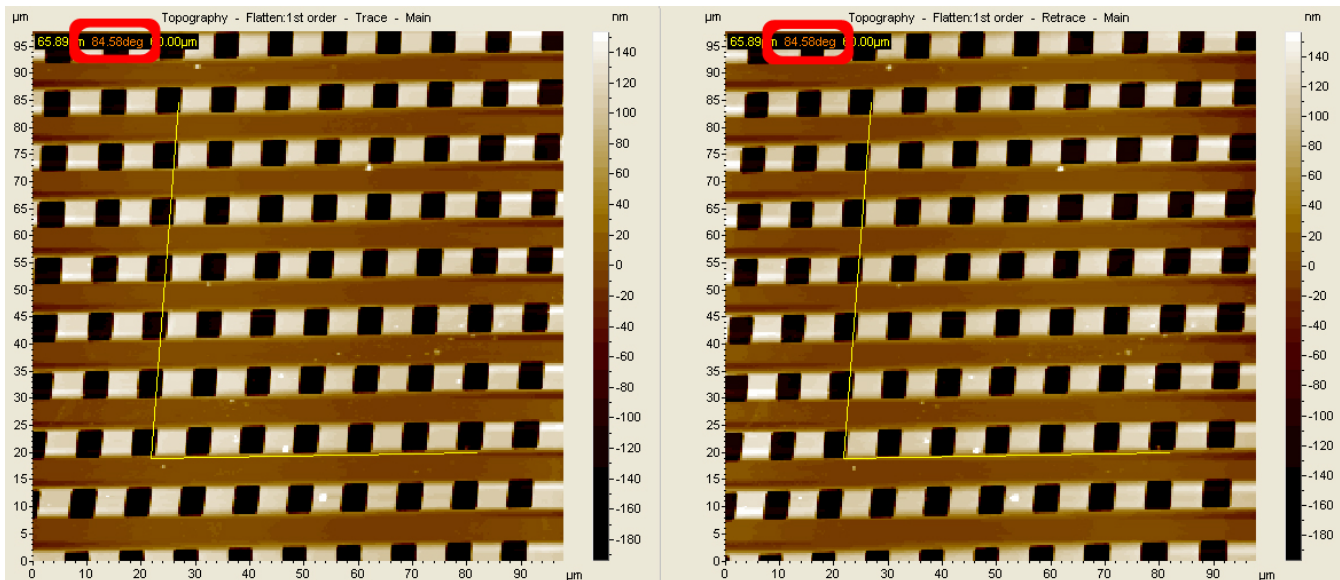


Figure 134 Angle measurement for calibrating Non-orthogonality

Adjust the Non-orthogonality term in the Open Loop tab of the Scanner Setup dialog box until the angle measures as close to 90 ° as possible for

all four measurements. An example of one of the corner angle measurements is shown below in Figure 135.

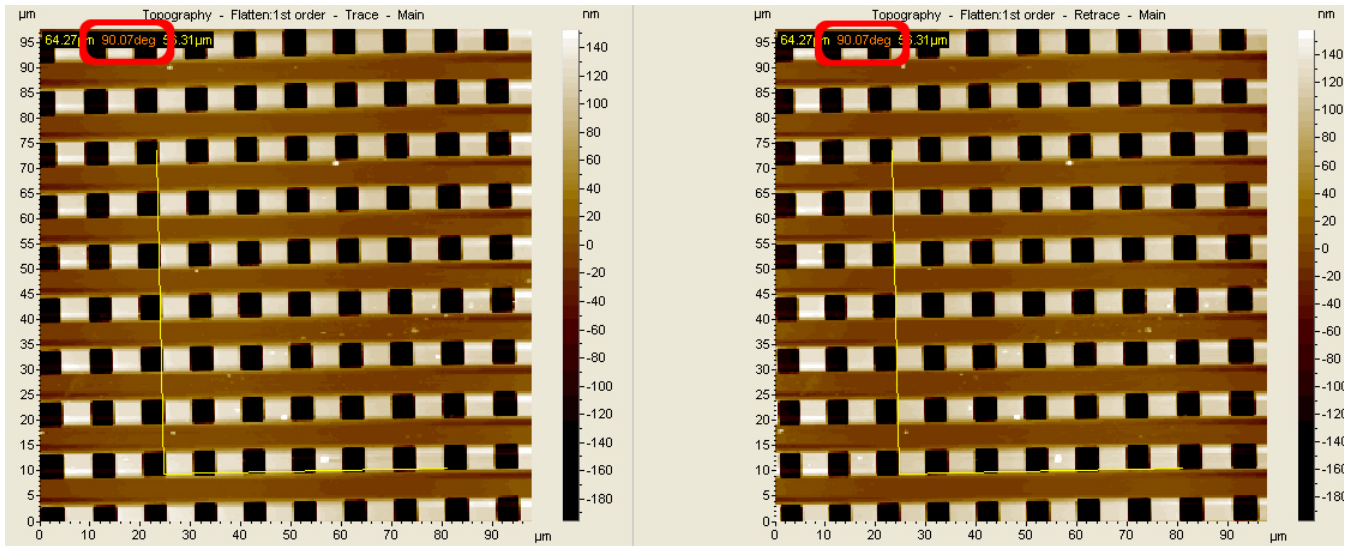


Figure 135 Angle measurement after calibrating Non-orthogonality

Z Calibration

Sensitivity

After the X and Y dimensions have been calibrated, obtain an image of a Z calibration standard and render the data as Tilted. Zoom in on a single pit to minimize distortion. Position the cross section tool over a Z feature. Place markers at the top and bottom of the feature. Measure the step size.

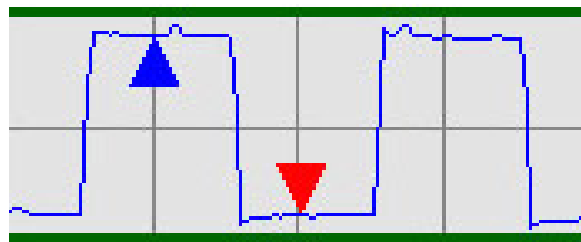


Figure 136 Cross-section checking feature height for Z sensitivity

It is important to correctly position the markers for the Z feature measurement. Place them in the center of the scan range. It may also be necessary to decrease the Integral (I) gain in the Main tab of the Servo dialog box until the top and the bottom of the data plot is flat before making the measurement.

If the step size is not within 5 % of the actual value, calculate a new Z sensitivity term using the following equation:

$$NewSensitivity = \frac{CurrentSensitivity \times KnownSize}{MeasuredSize}$$

After the X, Y and Z calibration steps have been completed, the scanner is fully calibrated for open loop operation. The remainder of this procedure will help to create and finalize the required calibration software files.

Servo Gain Multiplier

If you were to image a standard sample and view the topography or error signal on an oscilloscope while increasing the gains, you would see that, up to a point, the signal would be relatively smooth as the system accurately tracked the sample surface. At some gain level, however, the oscilloscope image will begin to display small, high frequency oscillations. This is similar to feedback in a microphone, with the gains so high that each oscillation is multiplied and fed back into the signal.

The Servo Gain Multiplier is a factor that sets the point at which this “feedback” will occur for a typical signal. Following a successful X, Y and Z calibration, the Servo Gain Multiplier must be set for each individual scanner. Enter the value in the Scanner Setup window. Experimentally it has been shown that an integral gain setting between 15 and 20 works well for most scanners.

Archive the Calibration Files

Copy the newly created calibration files to a disk for archive. Label the file with the scanner model, serial number and calibration date.

9 Closed-Loop Scanners

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In an open-loop scanner, a voltage is applied to the piezo actuators in extremely precise increments to move the probe accurately in all three axes. Nevertheless, inherent material properties of the piezo ceramics, such as hysteresis, creep, and aging may cause the piezoes to drift from these expected positions.

Keysight closed-loop scanners include high-precision, inductive positioning sensors to measure, compare, and correct the actual scanner position to the expected position. Closed-loop scanning improves scan linearity, provides more accurate probe positioning, and overcomes the effects of creep, hysteresis, etc.

Scanner Types

Two types of closed-loop scanners are available for the Keysight 5500 SPM: Z-axis closed-loop, and X/Y/Z closed-loop.

Z-Axis Closed-Loop Scanner

The Z-axis closed-loop scanner provides exceptional Z-axis positioning for very linear vertical positioning. Z-axis closed-loop is useful and more accurate when generating force curves as the sensor eliminates hysteresis and controls the piezo position to a much higher degree. Positional drift that may be present in an open-loop system is continuously corrected with the closed-loop sensor.

In the position tab of the Servo dialog box, you can select the **Set Z Position Using** check box to precisely maintain the cantilever at a

defined height above the sample for specialized experiments, such as measuring how long molecules stretch and relax (Figure 137).

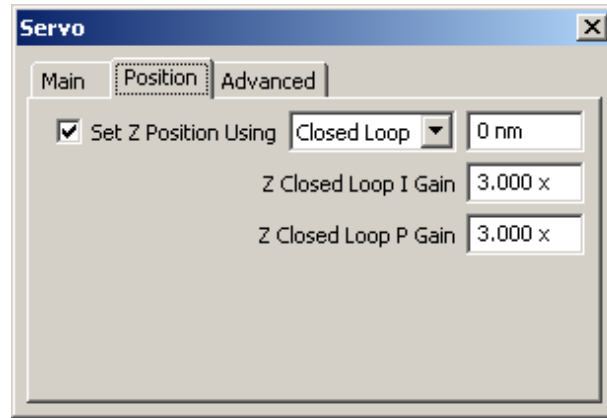


Figure 137 Setting Z position in the Position tab in the Servo dialog box

X/Y/Z Closed-Loop Scanner

The X/Y/Z closed-loop scanner includes the functionality of the Z-axis scanner described above, with additional encoders on the X and Y axes. These sensors allow for very linear scans and also make it easy to move to precise locations on the sample.

Calibration

Keysight recommends calibrating the Gains, Offset and Sensitivity settings in the Closed Loop tab of the Scanner Setup dialog box prior to each use of the closed-loop scanner. Begin by calibrating the scanner following the open-loop instructions in the [Chapter 8](#), "Scanner Maintenance and Calibration".

For a Z-axis scanner, skip to "[Z Sensor Calibration](#)" on page 206 below.

NOTE

The procedures and descriptions in this User Guide are for PicoView 1.12x and higher.

X and Y Sensor Calibration

Initial calibration of the X and Y sensors consists of matching the ± 10 V output range of the sensors with the actual piezo travel, which varies from scanner to scanner. No sample is required for this procedure.

- 1 Without a sample in place, load a cantilever and use the laser alignment controls and camera window to align the laser and detector mode to set up the microscope for **Contact Mode Imaging** as described in **Chapter 5**.
- 2 Choose **Controls > Setup > Controller** to confirm the appropriate controller definition file.
- 3 Choose **Controls > Setup > Microscope** to confirm the appropriate microscope definition file.
- 4 Verify that the correct scanner calibration file is selected in the Scanner menu.
- 5 Choose **Scanner > Edit** to open the Scanner Setup dialog box. Select the **Closed Loop** tab.

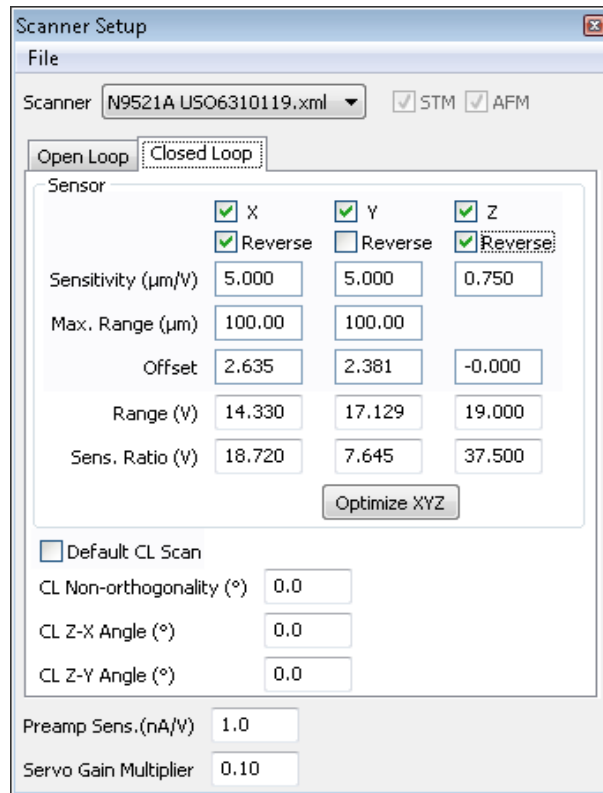


Figure 138 Scanner Setup dialog box

- 6 The values shown in [Figure 138](#) for the X, Y and Z Sensor, Offset, and Gain are typical.
 - a Ensure that the **Reverse** check boxes are selected for **X** and **Z** Sensors. If not, contact Keysight support.
 - b Note that the scanner values will be different for each scanner.
- 7 Verify that the **Default CL Scan** check box is **cleared** as shown above.
- 8 In the Advanced tab of the Scan and Motor window, set Overscan to **0 %** for Fast and Slow axes.

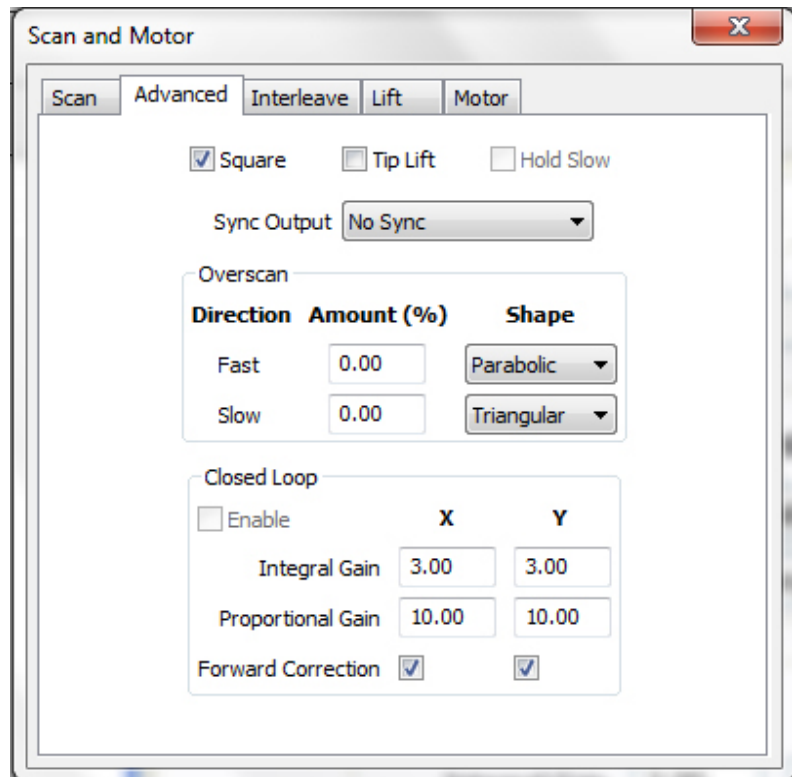


Figure 139 Advanced tab settings for CL calibration

- 9 Without a sample in place, use the laser alignment controls and camera window to align the laser and detector for Contact AFM mode.
- 10 In the Scan and Motor window enter:
 - a A very large number (e.g., 9999) for the Scan Size. It will adjust automatically to the maximum allowed value.
 - b Speed of **1-2 lines/second**.
 - c Resolution of **256**.
- 11 In the Realtime Images window, create two image buffers with the following settings:
 - Input set to **X Sensor**.
 - Flattening set to **None**.
 - Display Range set to **-10 V to +10 V**.
 - For one image display **Trace** and for the other display **Retrace**.
 - Set both buffers to **Main** scan mode.

- 12 Choose **Tools > Realtime Cross Section** so the individual scan lines will be displayed in the Cross Section window. Set the range to ± 10 V.
- 13 Choose **Controls > Trace/Retrace** and set the range to ± 10 V for the Trace/Retrace window.
- 14 Enable the high voltage by performing a false engagement:
 - a In the Servo Main tab, enter a Setpoint value more negative than the Deflection value displayed on the Head Electronics Box.
 - b Click the Approach button.
 - c Reduce the Z piezo Range to **0.000**.
 - d Click the **Center** button. This avoids the possibility that the piezo will become depolarized by being fully retracted (which is where it would be after the false engagement) for an extended period of time.
- 15 Click the **Up** or **Down** blue arrows in the Scan and Motor window to initiate a scan. When the scan is first started, the graph will probably look similar to that in [Figure 140](#).

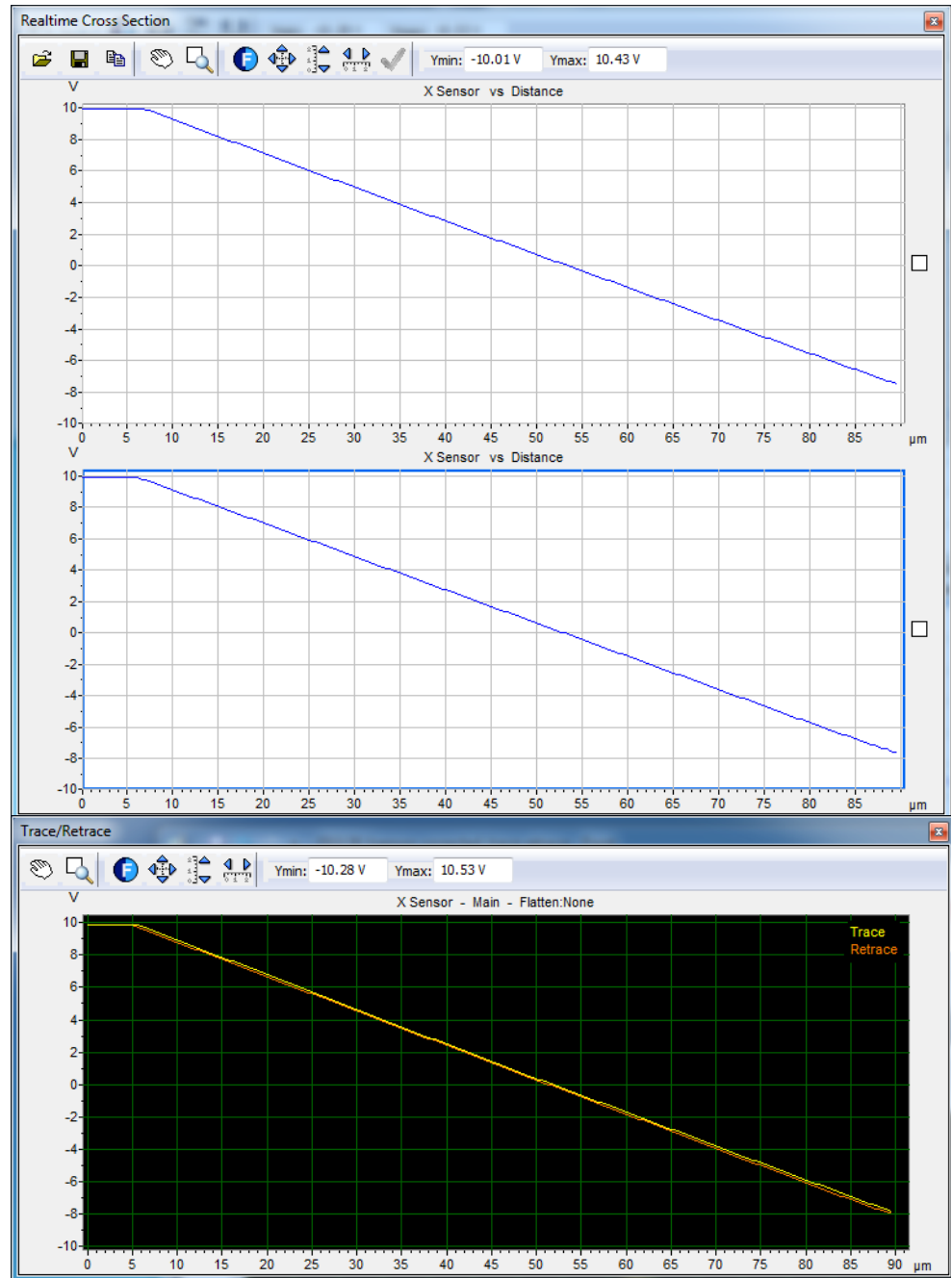


Figure 140 Initial plot before closed-loop scanner calibration

- 16 In the CL tab of the Scanner Setup dialog box, adjust the **X Sensor** values:
 - a Adjust the Offset to shift the line up or down until the left end is close to **-10 V**.
 - b Adjust the Gain to adjust the slope until the right end of the line is close to **+10 V**.

The cross-section graph should now appear as in Figure 141.

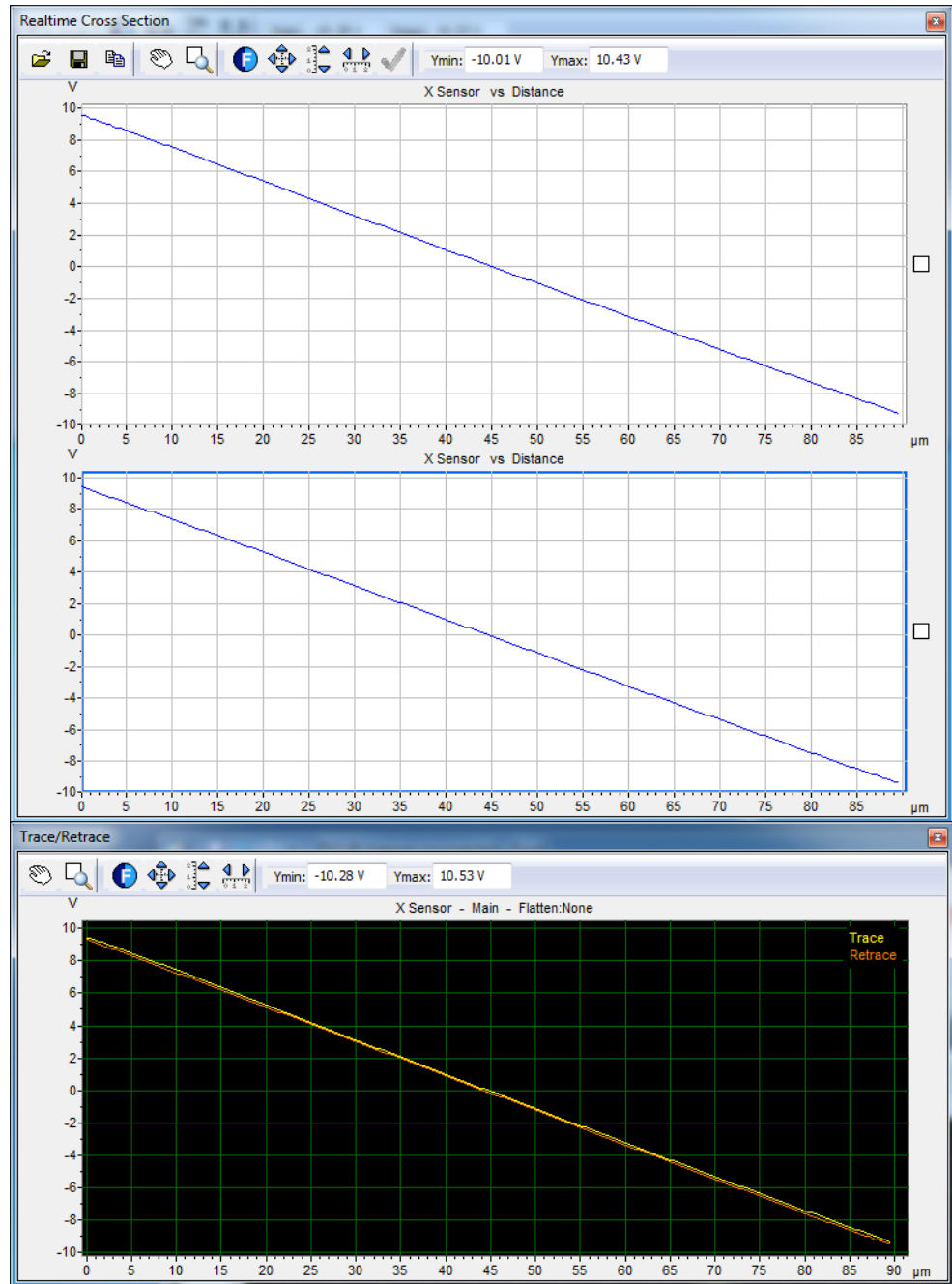


Figure 141 Cross Section and Trace/Retrace windows after Offset and Gain values optimized

- 17 In the Realtime Images window, change two image buffers to **Y Sensor**.
- 18 Use the Vertical Cross Section tool to display a Y cross-section.

- 19 Adjust the **Y Sensor** values:
- Adjust the Offset to shift the line up or down until the left end is close to **-10 V**.
 - Adjust the Gain to adjust the slope until the right end of the line is close to **+10 V**. Note this will take longer for the Y axis as is slower than the X axis.
- 20 In the CL section of the Advanced tab of the Scan and Motor window, select the **Enable** check box. The scanner will now function in closed-loop mode.
- 21 **Withdraw** the tip.
- 22 Save the new Scanner Setup parameters.
- In the Scanner Setup window, choose **File > Save As**.
 - Enter a filename.
 - Click **OK**.
- 23 Place a calibration grating on the standard sample plate and mount the plate on the microscope.
- 24 Align the grating in both the X and Y axes.
- 25 Perform all the Contact Mode imaging steps described in [Chapter 5](#) to achieve a good image of the grating.
- 26 Adjust the Sensitivity of the X sensor:
- In the Realtime Images window, choose **Tools > Horizontal Cross Section**. Place the cross-section tool across a set of features.
 - Set markers in the cross section window to measure the dimension across several features of known width.
 - Use the equation below to adjust the Sensitivity value:
- $$NewSensitivity = \frac{CurrentSensitivity \times KnownSize}{MeasuredSize}$$
- Image the features again and verify that the measured width matches the actual width.
- 27 Repeat the step above with the **Vertical Cross Section** tool to set the Sensitivity of the Y sensor.

The sensitivity ratio is the ratio of the closed loop sensor sensitivity to the open loop sensor sensitivity, and affects the forward correction feature. Open loop calibration can be performed for a small scan range

without affecting the agreement of trace and retrace in closed loop scanning.

28 Adjust the X sensor Sensitivity Ratio:

- a In the Realtime Images window, set up two Topography images, one for **Trace** and one for **Retrace**.
- b Place a vertical cursor at the same location in the Trace and Retrace images. The cursor should cross the same features in both images.
- c If this is not the case, adjust the X Sensitivity Ratio and re-scan. Alignment should be consistent across the full range of the X axis. Several iterations may be necessary to align all edges in the Trace and Retrace images.

29 Adjust the Y sensor Sensitivity Ratio:

- a In the Realtime Images window, assign one data channel to display a single frame of the calibration target and place a vertical cross-section through a line of features. Allow the scanner to scan continuously, which will update the Cross Section plot each pass through the frame.
- b Use the upward (trace) and downward (retrace) scan markers to measure the difference in the acquired data at a given point on the Y axis. While scanning in one direction, focus on one step of the grating. As the scan data is plotted, position a marker on this edge. Wait for the scan in the opposite direction to occur and position a second marker on the same edge after the plot has been updated. Alignment of the individual edges should be confirmed across the full range of the Y axis.
- c If this is not the case, adjust the Y Sensitivity Ratio and re-scan. Several iterations may be necessary to align all edges in both the upward and downward scans.

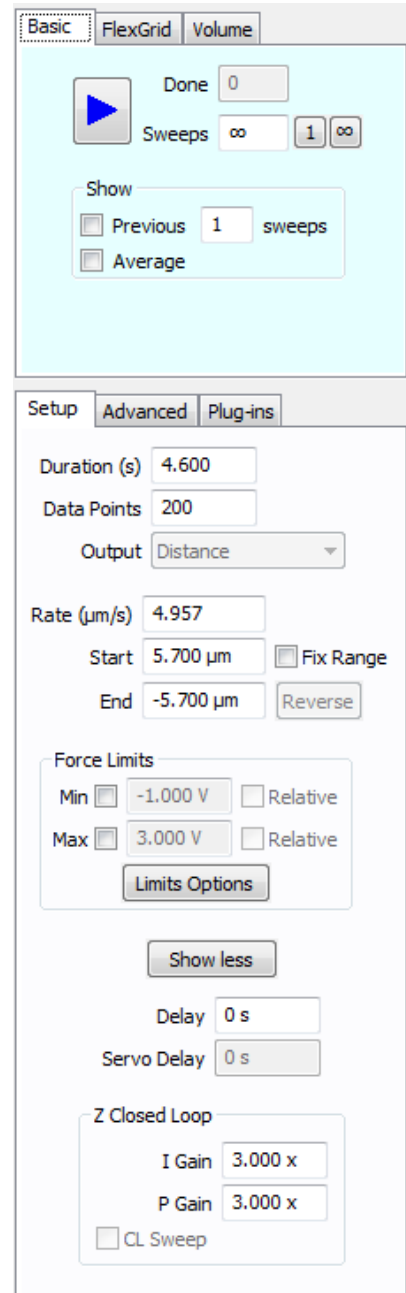
30 Save the Scanner Setup parameters.

Z Sensor Calibration

To calibrate the Z sensor, the output of the sensor will be plotted while the piezo is being moved through its entire range. Since only the motion

of the Z piezo is being measured, adjusting the Z Sensor Gain and Offset should be done without a sample in place.

- 1 In the Main tab of the Servo window, verify that the Z Range of the piezo is set to its maximum value.
- 2 Choose **Controls** > **Spectroscopy** to open the Spectroscopy window. Ensure two buffers are open.
- 3 In the Spectroscopy Basic tab, click the infinity ∞ button for continuous Sweeps.
- 4 In the Spectroscopy Setup tab, set Duration to **1**.
- 5 Set Data Points to **200**.
- 6 Maximize the Z piezo motion: maximize and minimize the **Start** and **End** values, respectively.
- 7 Click the **Show More** button to display more parameters.
- 8 Set the Gains to **3 x**.
- 9 In the Spectroscopy plot window, set the lower buffer to **Z Sensor**: go to the Data Channel for the plot and select from the drop-down menu as shown below in [Figure 142](#) on page 208.
- 10 Set the units to **V** from the drop-down menu in the plot window.



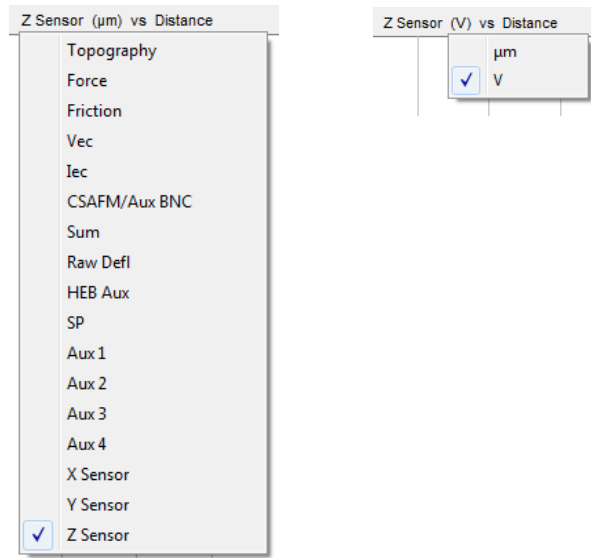


Figure 142 Select Z Sensor and units for Z sensor calibration

- 11 Click the blue triangle to start a continuous sweep of the Z piezo.
- 12 A warning message will be displayed. Click **Yes** to proceed with the calibration procedure.

The goal of the calibration procedure is to make the Z Sensor output appear like that shown in Figure 143.



Figure 143 Expected output for Z sensor calibration

The plot shows the output of the Z sensor as a function of Z piezo displacement. The Z sensor output ranges from -10 V to +10 V over the entire range of the Z piezo. This procedure sets the range just slightly under ± 10 V.

- 13 In the Spectroscopy plot window, turn Toggle Auto Full Scale **Off**.
- 14 Set Y Min to **-9.8** and Y Max to **+9.8**.
- 15 In the Z Sensor section of the Scanner Setup dialog box, adjust the **Offset** to change the vertical position of the lines until their left edge is as close to -9.8 V as possible.
- 16 Adjust the **Gain** to change the slope of the lines until they meet in the upper right corner.

NOTE

The Z piezo is highly sensitive to changes in Offset and Gain. To change these values in smaller increments, click the mouse in the Offset or Gain box. Hold down the **Ctrl** key while pressing the **Up** or **Down** arrows to change the parameter in 0.01 steps. Hold down the **Ctrl** and **Shift** keys while pressing the **Up** or **Down** arrow keys to change the values in 0.001 increments.

Once the plot looks like the example above in [Figure 143](#) on page 209, the Offset and Gain are properly set and the Z Sensor is usable over its entire range.

- 17 Select the **CL Sweep** check box. The plot should change from the one above to that shown in [Figure 144](#):

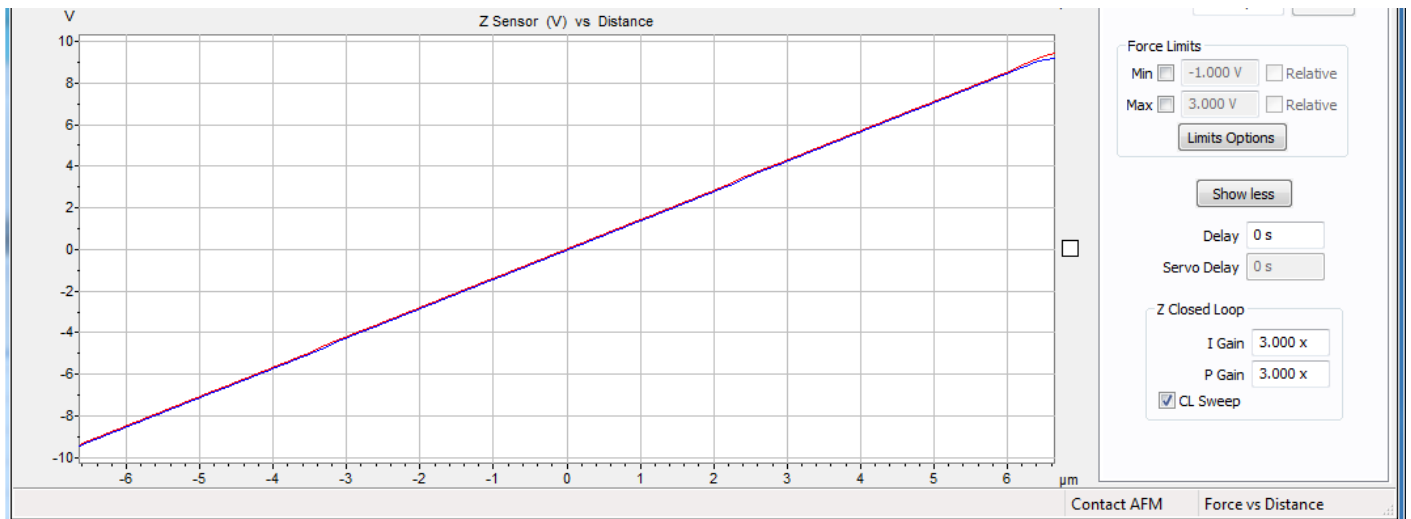


Figure 144 Spectroscopy sweep with closed loop On

- 18 In the Scanner Setup Closed Loop tab, adjust the Z Range setting to maximize the range. A typical starting value is 19 V.

CL Z Sensitivity

To calibrate the Z Sensitivity, you will need a step height calibration standard (the standard calibration grid supplied with your system will suffice). Its features are 200 nm deep.

- 1 Set up the microscope for Contact Mode imaging.
- 2 Initiate an approach.
- 3 Obtain a 10 micron image centered on one of the calibration standard pits (Figure 145). Flattening should be set to **None**.

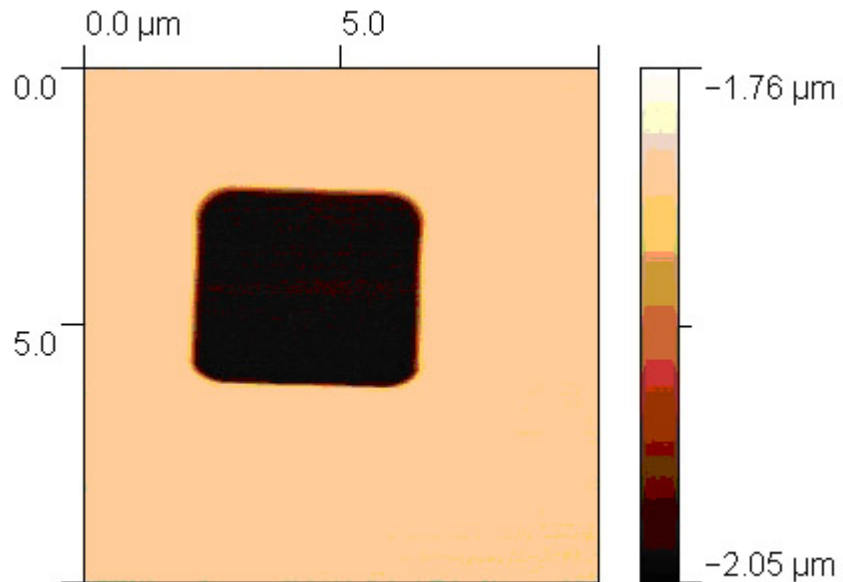


Figure 145 200 nm deep pit on the calibration standard

- 4 In the Realtime Images window, use a Horizontal or Vertical Cross Section tool to gauge a pit of known depth. If the depth does not match the actual value, calculate a new Sensitivity value using the following equation:

$$\text{NewSensitivity} = \frac{\text{CurrentSensitivity} \times \text{KnownSize}}{\text{MeasuredSize}}$$

- 5 Enter the new Z Sensitivity value into the Scanner Setup dialog box.
- 6 Image the same pit again and check that the depth is now measured at 200 nm. If not, repeat the previous steps until the value is measured correctly.

Save the calibration file.

X/Y/Z calibration is now complete.

10 MAC Mode

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The MAC Mode controller provides high precision AC signal control for AAC and MAC modes. The MAC controller uses a lock-in amplifier to generate the AC signal. It also provides routing capabilities and experimental controls for applications requiring additional flexibility in experiment design.

The MAC Mode controller is used in conjunction with the AFM Controller and Head Electronics Box (HEB). The main controller supplies high voltage to the scanner piezoes. The HEB controls the stage motors and receives information from the photodiode detector. The MAC controller supplies the drive signal to the nose assembly, as well as routing signals from additional inputs for advanced setups.

List of MAC Mode Components

The components you receive with MAC Mode may vary slightly depending on your purchased options:

- MAC Mode controller.
- DB44 cables.
- Power cable.
- RS-232 (serial) cable.
- AAC and/or MAC probes.
- Top MAC option (includes AAC and Top MAC nose assemblies, standard sample plate, 3-6 pin MAC cable, short DB44 cable) or MAC option (includes AAC nose assembly, MAC sample plate, 3-6 pin MAC cable, short DB44 cable).

Please contact Keysight if any of these items are missing.

Connections

The MAC Mode controller is shown in [Figure 146](#). The rear panel is shown in [Figure 147](#).



Figure 146 Front panel of the MAC Mode controller

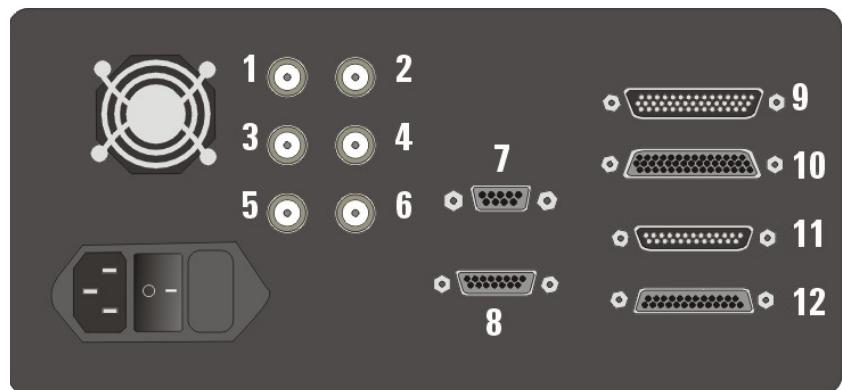


Figure 147 MAC Mode controller rear panel

The connectors are as follows:

- 1 MAC drive output.
- 2 AAC drive output.

- 3 Input summed to AAC drive signal.
- 4 Deflection output from detector.
- 5 Amplitude output from lock-in amplifier.
- 6 Phase shift signal from lock-in amplifier.
- 7 To Serial Port on computer.
- 8 Aux output for custom applications.
- 9 44-pin cable from AFM Controller.
- 10 44-pin cable to Head Electronics Box.
- 11 25-pin cable (if applicable).
- 12 25-pin cable to HEB (if applicable).

In addition to the standard cabling for your microscope, the following connections must be made to use the MAC Mode in your system (a complete wiring diagram is included in [Appendix A](#)).

Power Cord Connect the power cord from the back of the MAC Mode controller to building power. Do not power on the controller at this time.

Computer Connection Connect the RS-232 serial cable from the SERIAL port on the MAC Mode controller to a COM port on your computer. The port number will be automatically detected if your computer has more than one COM port.

Head Electronics Box Connection Connect the short DB44 cable from the MAC Mode Controller to the CONTROLLER connector on the Head Electronics box (HEB). Use a DB44 cable between the MICROSCOPE connector on the HEB and the 44-pin connector on the microscope.

AFM Controller Connection Connect a DB44 cable from the MAC Mode controller to the PicoSPM II connector on the AFM Controller.

Sample Plate Connection Plug the round jack of the EC/MAC cable into the underside of the microscope stand. Connect the MAC cable connector of the EC/MAC cable ([Figure 85](#) on page 125) to the 6-pin connector on the sample plate using the MAC cable ([Figure 86](#) on

page 126). When using Acoustic AC Mode, this connection is not necessary.

Hardware and Sample Setup

Most hardware and sample setup options with MAC Mode are identical to those for standard AAC and MAC Mode operation, as covered in [Chapter 6](#), “AC Modes.” Please refer to [Chapter 6](#) for more on how to set up the microscope for imaging. Note the phase conversion coefficient is 18 ° per volt for the MAC Mode controller.

In AAC Mode the drive signal can be provided by either the Head Electronics Box or the MAC Mode controller. To use the MAC Mode controller as the AC source, choose **Controls > Setup > Components**, then select the **Serial Port AC Mode Controller** check box. The system will now use the drive signal from the MAC Mode controller, through the HEB to the microscope.

Upon startup, the software may instruct you to update the system’s firmware. Follow the on-screen instructions to do so.

11

MAC III

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The MAC III controller adds imaging modes, routing capabilities and other experimental controls for applications requiring additional flexibility in experiment design. MAC III works with the Keysight 5500 SPM as either an option or an upgrade. It offers the best control available for oscillating probe technology, providing, among other things, far better resolution in fluids than other techniques.

MAC III includes three user-configured lock-in amplifiers for precise and versatile feedback options as well as additional experimental

flexibility. MAC III adds functionality to many of the imaging modes described earlier in this manual.

Moreover, MAC III can operate in multiple modes simultaneously. For example, you can image in AAC and KFM Modes simultaneously, ensuring that the same scan location and size is achieved in both modes. Not only is this arrangement very time efficient, it also ensures that the data is extremely reliable and easy to compare between modes.

The MAC III controller is used in conjunction with the AFM controller and Head Electronics Box (HEB). The AFM controller supplies high voltage to the scanner piezo elements. The HEB controls the stage motors and receives information from the photo detector. The MAC III controller supplies the drive signal to the nose assembly, as well as routing signals from additional inputs for advanced setups.

Initial Setup

List of MAC III Components

The components you receive with Mac III may vary slightly depending on your purchased options:

- MAC III controller.
- AAC nose assembly.
- DB44 cables.
- Power cable.
- RS-232 (serial) cable.
- MAC and/or AAC probes.
- Top MAC option (includes Top MAC nose assembly and standard sample plate) or MAC option (includes MAC mode sample plate and 3-6 pin MAC cable).

Please contact Keysight if any of these items are missing.

Connections

The MAC III controller is shown in [Figure 148](#). The rear panel is shown in [Figure 149](#).



Figure 148 Front panel of the MAC III Controller.



Figure 149 Rear panel of the MAC III Controller.

In addition to the standard cabling for your microscope, the following connections must be made to use the MAC III in your system. A complete wiring diagram is available in [Appendix A](#).

Power Cord Connect the power cord from the back of the MAC III controller to building power. Do not power on the controller at this time.

Computer Connection Connect the RS-232 serial cable from the SERIAL port on the MAC III controller to a COM port on your computer. The port number will be automatically detected if your computer has more than one COM port.

Head Electronics Box Connection Connect the shorter DB44 cable from the MICROSCOPE connection on the MAC III controller to the CONTROLLER connector on the Head Electronics box (HEB). The HEB is meant to be placed on top of the MAC III box. Use a DB44

cable between the MICROSCOPE connector on the HEB and the 44-pin connector on the microscope base.

AFM Controller Connection Connect the longer DB44 cable from the CONTROLLER connector on the MAC III to the PicoSPM II connector on the AFM Controller.

BNC 1 and 2 These connectors are user configured outputs for custom applications.

AUX The AUX connector has the drive output from each lock-in, a drive-in that can be summed into each lock-in, and an auxiliary input to each lock-in. The pin-out diagram is shown in **Figure 150**:

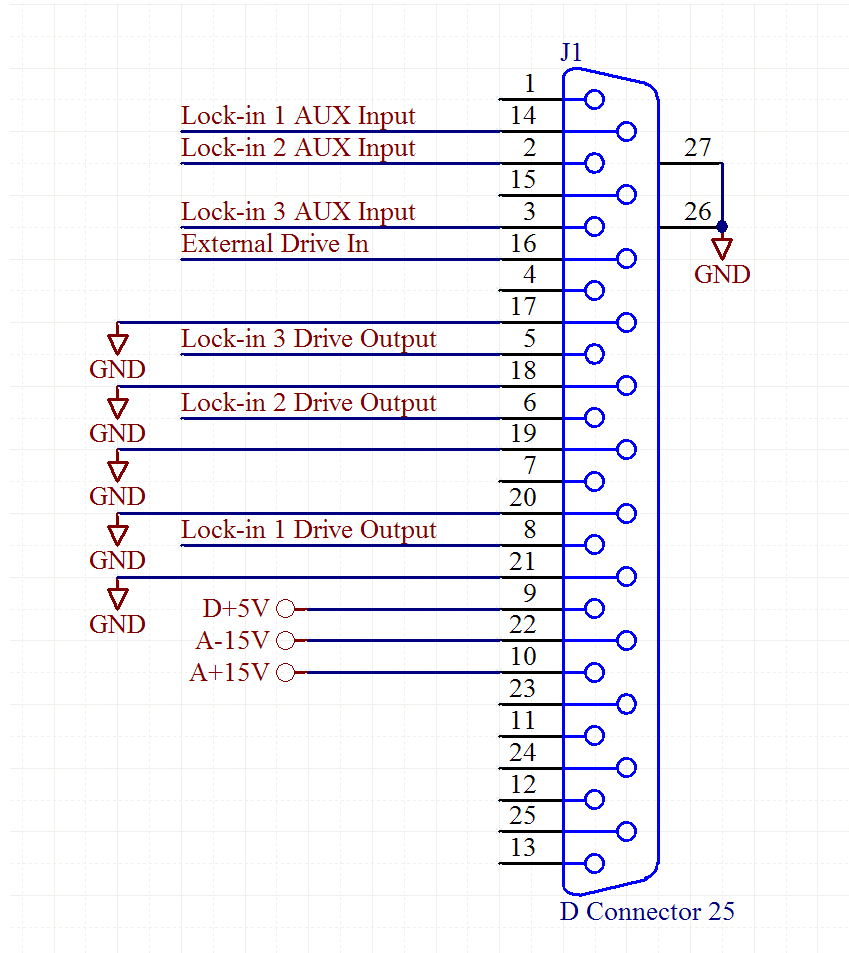


Figure 150 AUX Connector pin-out diagram

In this diagram the numbers refer to the slots in which the lock-in cards sit inside the MAC III box. Lock-ins 1, 2 and 3 are located in slots 1, 3 and 5, respectively. The connections are as follows:

AUXIN 1-5	AUX inputs for each slot. AUXIN 1, 3 and 5 are the AUX inputs for Lock-ins 1, 2 and 3, respectively.
Drive 1-5	The drive outputs from each slot. DRIVE 1, 3 and 5 are the Drive Out signals for Lock-ins 1, 2 and 3, respectively.
Drive_In	A single drive line that can optionally be summed in to any or all of the drives by using Sum External Drive on the Lock-in tab of the Advanced AC Modes window.
SP_RX and SP_TX lines	These serial lines are not currently used.

Once all connections have been made it is safe to turn on power to all components.

Hardware and Sample Setup

Most hardware and sample setup options with MAC III are identical to those for standard AAC and MAC Mode operation, as covered in [Chapter 6](#), “AC Modes.” Please refer to [Chapter 6](#) for the steps required to set up the microscope for imaging. Note the phase conversion coefficient is 18 ° per volt for the MAC III controller.

Auxiliary Signal Access Box

An optional signal access box is available for MAC III to allow custom multi-frequency experimentation. The Auxiliary Signal Access Box (ASAB) can be used in the development of novel capabilities for the examination of local mechanical and electromagnetic properties. The box is shown in [Figure 151](#) and described in [Table 4](#). Specific examples

of EFM and KFM applications using the box are detailed in "Amplitude and Frequency Modulation Techniques" on page 249.



Figure 151 Optional Auxiliary Signal Access Box for MAC III

The ASAB provides access to the three lock-in amplifiers on the MAC III Controller or AAC III Controller through the 25-pin AUX connector.

Table 4 List of signals and descriptions for MAC III Signal Access Box

Signal	Description
External Drive Input	This is an external input signal in the range of ± 10 V that can be added to any of the drive outputs. To enable summing of this external drive input to any of the lock-in drive outputs, open the Advanced AC Mode Controls window, select the appropriate tab of the lock-in amplifier and select the Sum External Drive check box.

<p>Lock-in 1 Drive Output</p>	<p>This is the drive output of Lock-in Amplifier 1 and can be used as the AC stimulus of an external experiment. The output is nominally in the range of ± 10 V. A DC bias can also be added by entering a voltage for the Drive Offset in the Lock-in 1 tab in the Advanced AC Mode Controls window.</p>
<p>Lock-in 1 Input</p>	<p>This is the auxiliary input of Lock-in Amplifier 1. The nominal input range is 2.5 V peak to peak AC within the range of ± 10 V DC. To use this input to Lock-in 1, open the Advanced AC Mode Controls window, select the Lock-in 1 tab, then select Aux from the Input dropdown menu.</p> <p>This can also be used as an external input to the controller. While this is in use it disables the normal outputs of the Lock-in. To use this as an external input to the controller, select the Lock-in 1 tab in the Advanced AC Mode Controls window and select the Y Component From Aux check box to enable the Auxiliary signal to replace the Y component. Then in the Output tab, select Y Component 1 as an output to one on the controller auxiliary inputs.</p>
<p>Lock-in 2 Drive Output</p>	<p>This is the drive output of Lock-in Amplifier 2 and can be used as the AC stimulus of an external experiment. The output is nominally in the range of ± 10 V. A DC bias can also be added by entering a voltage for the Drive Offset in the Lock-in 2 tab in the Advanced AC Mode Controls window.</p>
<p>Lock-in 2 Input</p>	<p>This is the auxiliary input of Lock-in Amplifier 2. The nominal input range is 2.5 V peak to peak AC within the range of ± 10 V DC. To use this input to Lock-in 2, open the Advanced AC Mode Controls window, select the Lock-in 2 tab, then select Aux from the Input dropdown menu.</p> <p>This can also be used as an external input to the controller. While this is in use it disables the normal outputs of the Lock-in. To use this as an external input to the controller, select the Lock-in 2 tab in the Advanced AC Mode Controls window and select the Y Component From Aux check box to enable the Auxiliary signal to replace the Y component. Then in the Output tab, select Y Component 2 as an output to one on the controller auxiliary inputs.</p>
<p>Lock-in 3 Drive Output</p>	<p>This is the drive output of Lock-in Amplifier 3 and can be used as the AC stimulus of an external experiment. The output is nominally in the range of ± 10 V. A DC bias can also be added by entering a voltage for the Drive Offset in the Lock-in 3 tab in the Advanced AC Mode Controls window.</p>

<p>Lock-in 3 Input</p>	<p>This is the auxiliary input of Lock-in Amplifier 3. The nominal input range is 2.5 V peak to peak AC within the range of ± 10 V DC. To use this input to Lock-in 3, open the Advanced AC Mode Controls window, select the Lock-in 3 tab, then select Aux from the Input dropdown menu.</p> <p>This can also be used as an external input to the controller. While this is in use it disables the normal outputs of the Lock-in. To use this as an external input to the controller, select the Lock-in 3 tab in the Advanced AC Mode Controls window and select the Y Component From Aux check box to enable the Auxiliary signal to replace the Y component. Then in the Output tab, select Y Component 3 as an output to one on the controller auxiliary inputs.</p>
-------------------------------	---

Connections for ASAB

There are two configurations for connecting the ASAB to the MAC III controller. One is for Frequency Modulation (FM) EFM and KFM. The other is for Piezoresponse Force Microscopy (PFM) experiments.

For applying EFM and KFM FM modes, Lock-in 1 is used for the Z servo, Lock-in 2 is drives the tip or sample bias, and Lock-in 3 can be used to monitor second or third harmonics for the Lock-in 2 frequency.

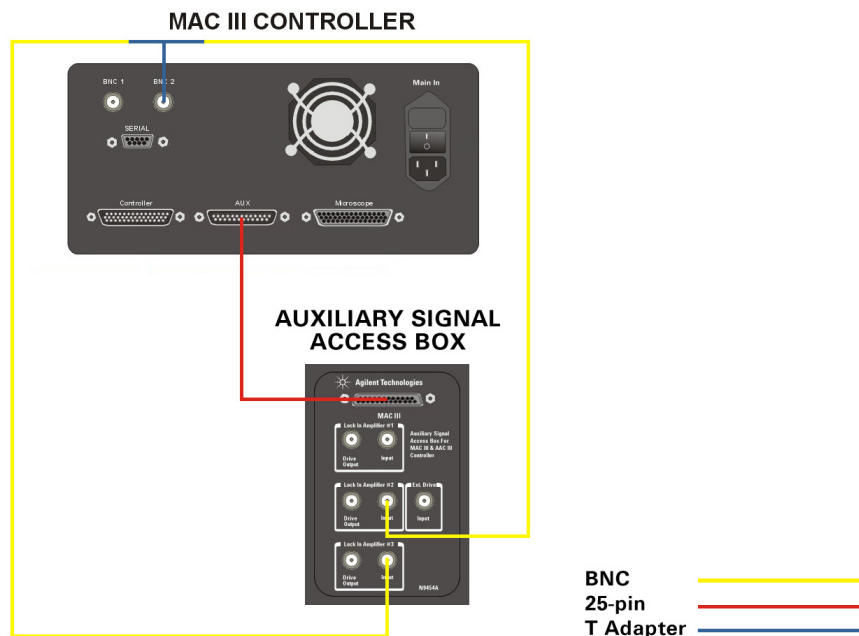


Figure 152 Wiring diagram for FM-KFM mode

First connect the 25 pin cable between the ASAB MAC III Aux connector to the MAC/AACIII Aux Connector. This is shown as the red line in [Figure 152](#) on page 225.

For the minimum configuration with no harmonics for KFM and EFM FM modes, add one BNC cable between ASAB Lock-in 2 Input and the MAC III or AAC III controller BNC2.

For the full configuration, designated by the yellow and blue lines in [Figure 152](#) on page 225, add a BNC “T” adapter to the MAC III or AAC III controller BNC2 and connect two BNC cables:

- 1 to the ASAB Lock-in Amplifier 2 Input and
- 2 to the ASAB Lock-in Amplifier 3 Input.

In PFM mode, Lock-in 2 drives the sample bias and monitors the deflection signal to measure the amplitude of the cantilever motion caused by the piezoelectric motion of the sample. This motion is very small and often requires a very high gain before the lock-in (total Gain x1024). The bias voltage applied between the tip and sample is usually very large (20 Volts peak to peak) and the AC bias signal is routed separately.

For PFM Mode, first connect the 25 pin cable between the ASAB MAC III Aux connector to the MAC/AACIII Aux Connector. This is shown as the red line in [Figure 153](#) on page 227.

Next connect a 6 foot BNC cable from ASAB Lock-in Amplifier 2 Drive Output to the sample plate connector adapter, as shown by the

yellow line in Figure 153. Then connect the adapter to the sample plate. Be sure the conductive substrate is connected to the sample bias.

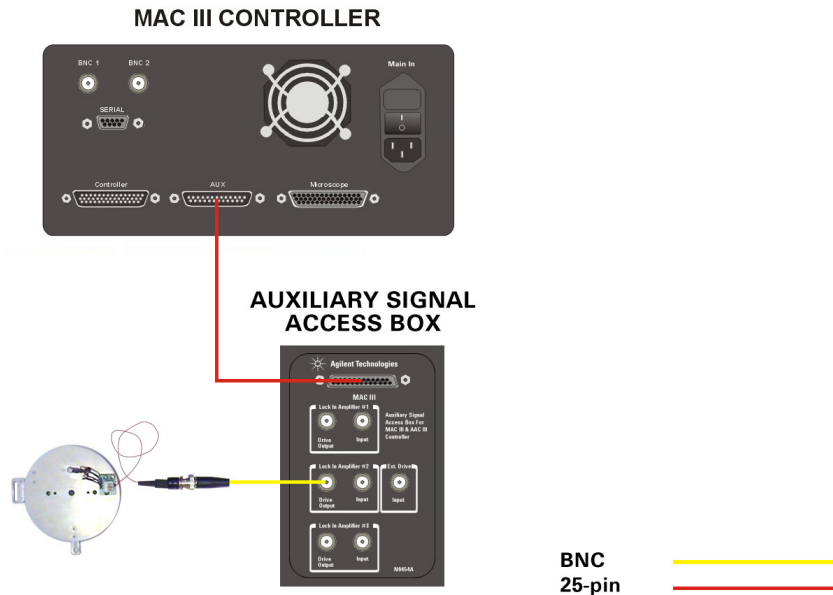


Figure 153 Wiring diagram for Piezoresponse Force Microscopy experiments

MAC III Software Controls

PicoView software provides two ways to access and control the various imaging modes.

Simplified Controls From the **Mode** menu, select the imaging mode you wish to use. PicoView will open a window with only the options required for that mode and will automatically adjust settings to typical values for that mode. This is the way that you will most often use MAC III.

Advanced Controls The **Controls > Advanced > AC Mode** window displays all of the possible MAC III options. This window is used to create custom imaging setups,

or when more control is needed over a predefined mode.

NOTE

The procedures and descriptions in this User Guide are for PicoView 1.10x and higher.

Simplified Software Control Options

Selecting an operating mode from the Mode menu will provide a window with just the controls needed for that mode and will automatically adjust parameters to appropriate values. The changes will also be visible in the Advanced AC Mode property sheet.

In this section we will describe the simplified controls for each mode.

Contact Mode

Contact mode does not require any MAC III-specific controls.

AC AFM

In AC mode the drive signal can be provided by the MAC III controller. To use the MAC III controller as the AC source choose **Controls > Setup > Components**, then select the **Serial Port AC Mode Controller** check box. The system will now use the drive signal from the MAC III controller, through the HEB to the microscope. Whenever

the MAC III controller is connected, this option should be selected to ensure proper operation.

AC Mode monitors and controls the oscillation amplitude of the cantilever. Choosing **Controls > AC Mode** opens the ACAFM Controls window:

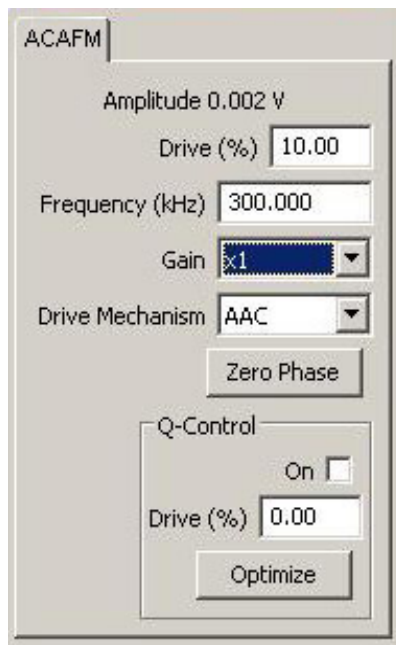


Figure 154 ACAFM Controls window

In AC Mode, Lock-in 1 is enabled by default, providing an oscillating signal to drive the cantilever. The Deflection channel is selected as the input for Lock-in 1 during laser and detector alignment. When an approach is initiated, the input automatically switches to Amplitude.

The following parameters are available in the ACAFM Controls window:

Amplitude	Displays the amplitude, in volts, of cantilever oscillation.
Drive (%)	The amplitude of the lock-in drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the lock-in signal. From the AC Tune window you can sweep the

frequency of Lock-in 1 to determine the resonance frequency of the cantilever.

Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding 10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).
Drive Mechanism	The mechanism (AAC, MAC or Top MAC) used on the microscope.
Zero Phase	Sets the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Optimize	Sets the optimal Q-Control Phase Shift and Drive.

When you initiate the approach, the Amplitude 1 and Phase 1 signals will be routed through the Deflection and Friction channels, respectively, to the main controller. During the laser and detector alignment procedure the **Pass Through** check boxes will be selected to

allow the correct signals to pass through to the Laser Alignment window.

STM

STM does not require any MAC III-specific controls.

LFM

Contact mode does not require any MAC III-specific controls.

DLFM

In DLFM mode, Lock-in 1 is used to oscillate the tip parallel to the sample surface, with the Friction channel as its input. The Drive Mechanism is set to **AAC**. Cantilever deflection is controlled as in Contact Mode.

CAUTION

The piezo element used to oscillate the cantilever in the DLFM nose assembly is partially exposed; therefore, DLFM should **never** be used for imaging in liquid.

Choose **Mode > DLFM**, then choose **Controls > AC Mode** to open the DLFM Controls window:

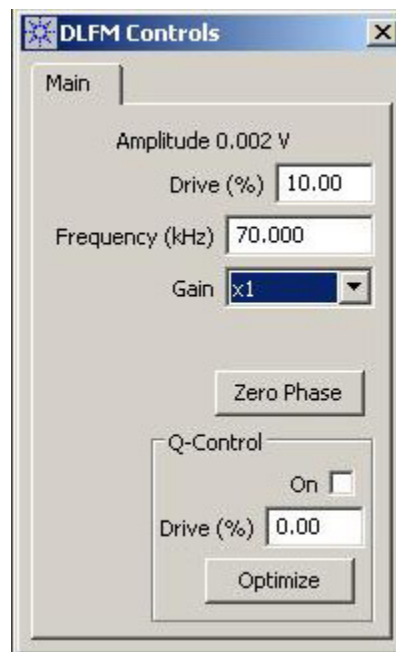


Figure 155 DLFM Controls window

Amplitude	Displays the amplitude, in volts, of cantilever lateral oscillation amplitude.
Drive (%)	The amplitude of the lock-in drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the lock-in signal. From the AC Tune window you will be able to sweep the frequency of Lock-in 1, to determine the frequency at which the lateral tip deflection is maximized (i.e., the resonant frequency).
Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding 10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).
Zero Phase	Sets the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Optimize	Sets the optimal Q-Control Phase Shift and Drive.

Amplitude 1 and Phase 1 are routed to the Aux 1 and Aux 2 outputs, respectively. These signals can be viewed by selecting Aux 1 or Aux 2 from the drop-down list in the Realtime Images window.

FMM

In Force Modulation Mode, Lock-in 1 provides the oscillating signal driving the cantilever. The amplitude of cantilever modulation that

results from this applied signal is monitored as a measure of the sample's elastic properties.

Force Modulation is a contact imaging mode and the Deflection signal will be routed to the feedback loop.

Choose **Mode > Force Modulation**, then choose **Controls > AC Mode** to open the Force Modulation Controls window:

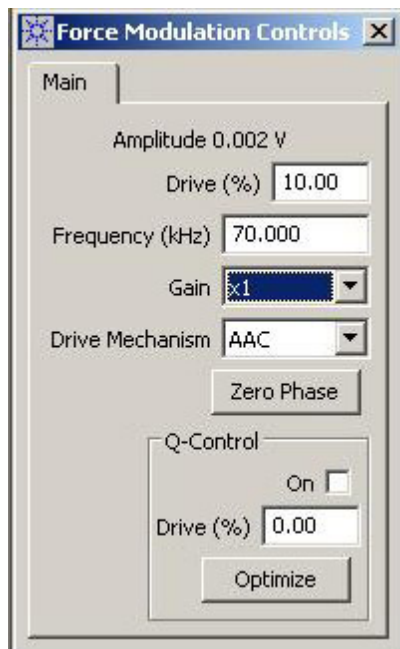


Figure 156 Force Modulation Controls window

In Force Modulation Mode, Lock-in 1 is enabled, with the Deflection channel as its input. The following settings are also available:

Amplitude	Displays the amplitude, in volts, of cantilever deflection.
Drive (%)	The amplitude of the lock-in drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the lock-in signal.
Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding

10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).

Drive Mechanism	The mechanism (AAC, MAC or Top MAC).
Zero Phase	Sets the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Optimize	Sets the optimal Q-Control Phase Shift and Drive.

On the Output tab, the **Pass Through** check boxes for Deflection and Friction are selected, passing these values from the microscope through to the AFM controller.

Amplitude 1 and Phase 1 are routed to the Aux 1 and Aux 2 outputs, respectively, to be used as inputs to image buffers. These signals can be viewed by selecting Aux 1 or Aux 2 from the drop-down list in the Realtime Images window.

EFM

In EFM Mode, Lock-in 1 is used to drive the cantilever, with the Deflection channel as its Input. Lock-in 2 provides an AC tip bias, also with the Deflection channel as its Input. The actual Deflection input during scanning is the oscillation amplitude.

NOTE

For EFM mode, the Bias switch on the back of the Head Electronics Box must be set to **Tip**.

Choose **Mode > EFM**, then choose **Controls > AC Mode** to open the EFM Controls window:

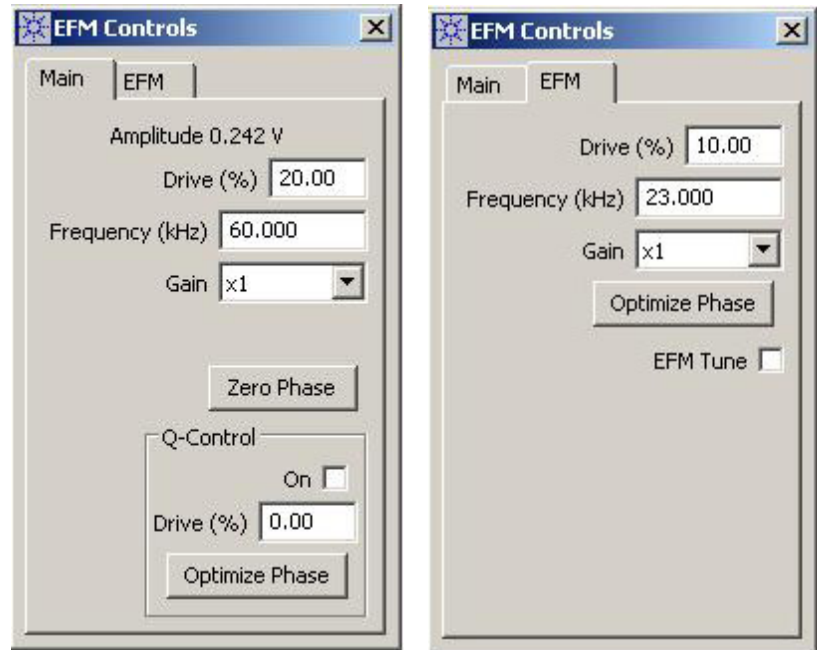


Figure 157 EFM Controls window

The Main tab includes settings for Lock-in 1 and Q-Control:

Amplitude	Displays the amplitude, in volts, of cantilever oscillation amplitude.
Drive (%)	The amplitude of the Lock-in 1 drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the Lock-in 1 signal. From the AC Tune window you can sweep the frequency of Lock-in 1, to determine the frequency at which the tip oscillation is maximized (i.e., the resonant frequency).
Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding

	10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).
Zero Phase	Sets the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Optimize	Sets the optimal Q-Control Phase Shift and Drive.

The EFM tab shows the parameters for Lock-in 2. Drive, Frequency and Gain settings have the same functions as described for Lock-in 1 in "[Lock-In Tabs](#)" on page 241.

As mentioned, Lock-in 2 is used as the source for the AC tip bias. You will typically need to sweep the frequency of Lock-in 2 to ensure that the electrical response of the cantilever does not interfere with the mechanical response provided by Lock-in 1 and to see if there are any other resonances present. To do so, select the **EFM Tune** check box, then choose **Manual Tune** in the AC Tune window. Determining the best frequency for your sample and tip will require some iteration. Two rules typically apply:

- The frequency should not be an integral factor of the Lock-in 1 frequency.
- The frequency should not be close (within 10-20 kHz) to the Lock-in 1 frequency.

Optimize shifts the phase signal to maximize the X Component 2 (i.e., to maximize contrast).

X Component 2 and Phase 2 are routed to the Aux 1 and Aux 2 outputs, respectively, for monitoring. To view changes in the EFM signal, choose Aux 1 in the Realtime Images window.

KFM

In KFM Mode, Lock-in 1 is used to drive the cantilever, with the Deflection channel as its Input. Lock-in 2 provides an AC tip bias, also with the Deflection channel as its Input. A DC bias is provided by an internal servo mechanism to counter electrostatic forces on the tip.

NOTE

For KFM mode, the Bias switch on the back of the Head Electronics Box must be set to **Tip**.

Choose **Mode > KFM**, then choose **Controls > AC Mode** to open the KFM Controls window:

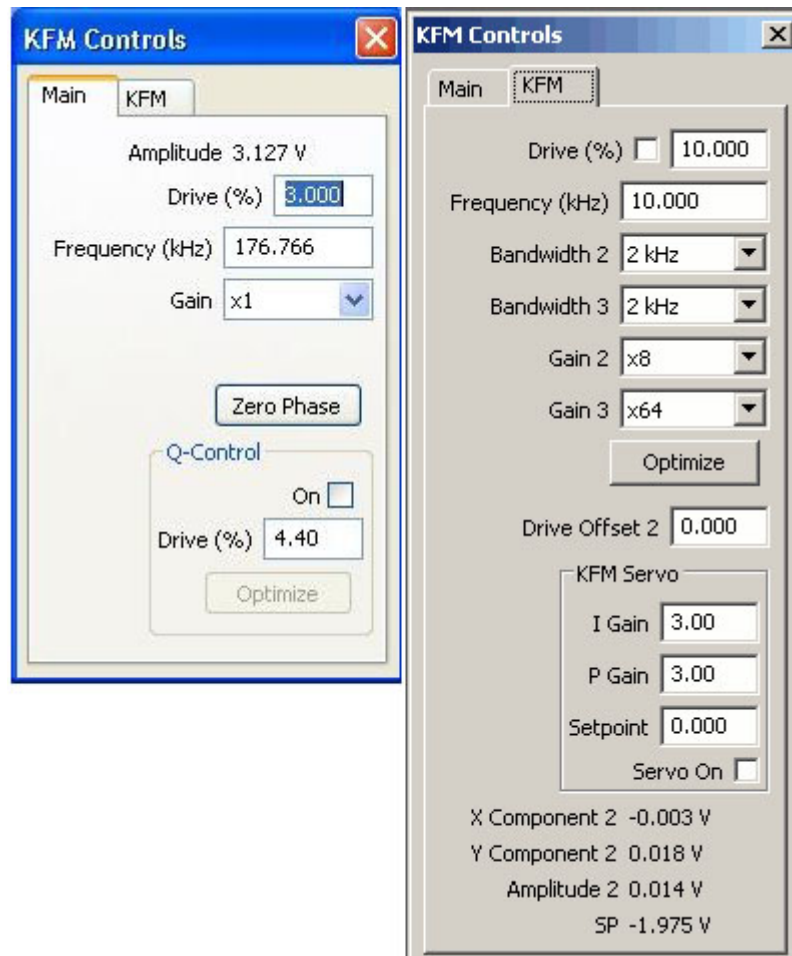


Figure 158 KFM Controls window

The Main tab shows settings for Lock-in 1 and Q-Control:

Amplitude	Displays the amplitude, in volts, of cantilever oscillation amplitude.
Drive (%)	The amplitude of the Lock-in 1 drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the Lock-in 1 signal. From the AC Tune window you can sweep the frequency of Lock-in 1, to determine the

	frequency at which the tip oscillation is maximized (i.e., the resonant frequency).
Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding 10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).
Zero Phase	Sets the phase at the current frequency to zero, making it easier to interpret phase changes from the current value.
Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Optimize	Sets the optimal Q-Control Phase Shift and Drive.

The KFM tab shows the parameters for Lock-in 2. **Drive**, **Frequency**, **Bandwidth** (Lock-in 2 and 3) and **Gain** (Lock-in 2 and 3) settings have the same functions as described for Lock-in 1 in "[Lock-In Tabs](#)" on page 241.

As mentioned, Lock-in 2 is used as the source for the AC tip bias. You will typically need to sweep the frequency of Lock-in 2 to ensure that the electrical response of the cantilever does not interfere with the

mechanical response provided by Lock-in 1 and to see if there are any other resonances present.

To do so, select the **KFM Servo** check box, then choose **Manual Tune** in the AC Tune window. Determining the best frequency for your sample and tip will require some iteration. Two rules typically apply:

- The frequency should not be an integral factor of the Lock-in 1 frequency.
- The frequency should not be close (within 10-20 kHz) to the Lock-in 1 frequency.

Optimize shifts the phase signal to maximize the X Component 2 (i.e., to maximize contrast).

The output from the servo is routed to the SP Channel and to the Drive Offset of Lock-in 2. To map the output (which is the KFM signal), choose **SP** in the Realtime Images window.

I Gain and P Gain are the Integral and Proportional Gains for the MAC III internal servo loop. Set the I and P Gains to obtain the sharpest image in the Realtime Images window.

X Component 2 and Phase 2 are routed to the Aux 1 and Aux 2 outputs, respectively, for monitoring. To view changes in the KFM signal, choose Aux 1 in the Realtime Images window.

Advanced Software Control Options

The Advanced AC Mode property sheet gives you more signal routing and control options than the simplified options described above. To view the AC Mode settings, choose **Controls > Advanced > AC Mode**.

The Advanced AC Mode window includes several tabs, each of which is described below.

Lock-In Tabs

Each of the three lock-ins includes its own tab with the following settings:

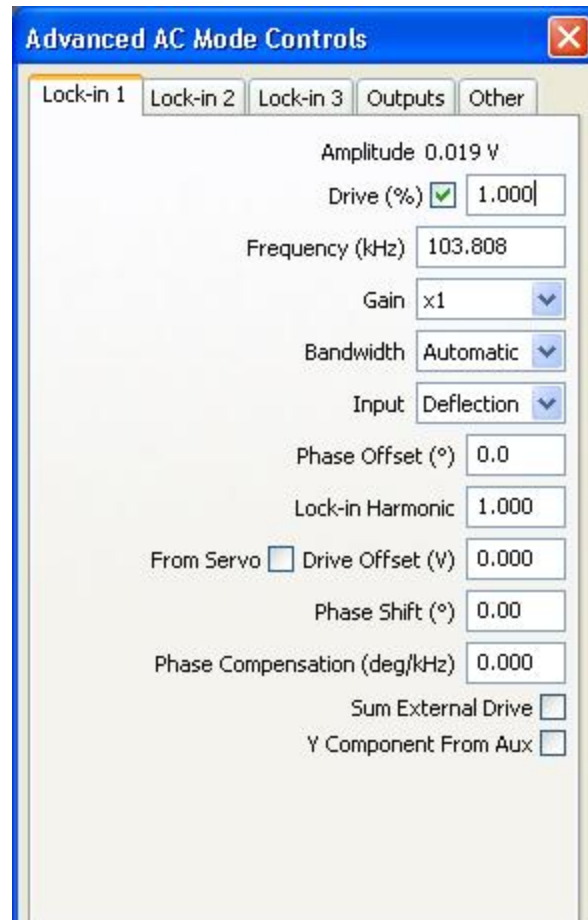


Figure 159 Advanced AC Mode Controls window: Lock-in tab

Amplitude

Displays the amplitude, in volts, of whatever is being driven by the lock-in drive. For example, if the lock-in is driving the

	cantilever, the oscillation amplitude (as measured by the photo detector) is reported.
Drive (%)	The amplitude of the Lock-in 1 drive signal, stated as a percentage (0-100 %) of the maximum available 10 V.
Frequency (kHz)	Displays the frequency of the Lock-in 1 signal. From the AC Tune window you can sweep the frequency of Lock-in 1, to determine the frequency at which the tip oscillation is maximized (i.e., the resonant frequency).
Gain	Multiplies the output of the lock-in by the selected factor. Use a larger multiplier to improve the signal-to-noise ratio for a small signal. Ensure that the gain will not result in an amplitude exceeding 10 V, beyond which the signal will be clipped. The default value is x1 (the amplitude times 1).
Bandwidth	How far to either side of the selected Frequency the lock-in circuitry is able to process information. Bandwidth can range from 80 Hz to 20 kHz. The 80 Hz detection bandwidth is ± 40 Hz about the center frequency. The default "Automatic" setting will adjust the bandwidth based on the Input signal.
Input	The source signal that is routed to the input of the lock-in. Choosing Aux will route the signal from the MAC III controller's AUX connector to the lock-in input. The default Input is the cantilever Deflection.
Phase Offset	Applies an offset to the calculated phase signal. The value, stated in degrees, is 0 by default; however, it can be adjusted such that the calculated phase will read 0, making it easier to interpret changes in phase. The Auto Tune function (Controls > AC Mode Tune) will automatically set this offset value.
Lock-In Harmonic	Setting this value will drive the reference signals at a fraction or multiple of the drive

	<p>signal. This value allows you to examine the signal at harmonics of the drive signal. The default value is 1.</p>
Drive Offset	<p>Applies an offset, in volts, to the drive signal. The default value is 0.</p> <p>Select the From Servo check box to add the output from the MAC III internal servo loop to the drive signal. This option is used in KFM Mode, in which the servo acts to maintain a DC Tip Bias that counteracts any electrostatic field on the sample. The servo output, therefore, will change as the sample charge changes; this value is also fed to the SP channel for imaging.</p>
Phase Shift (°)	<p>Each lock-in includes two orthogonal reference signals. This parameter will shift the phase of the reference signals with respect to the drive signal (they will remain orthogonal to each other). This feature is useful, for example, to maximize the real component of the drive signal in KFM mode. The default value is 0.</p>
Phase Compensation	<p>Phase Compensation adds a frequency dependent phase offset to compensate for any phase shifts in the electronics.</p>
Sum External Drive	<p>Select this check box to add a signal from the ASAB External Drive Input to the lock-in drive signal. By default the check box cleared.</p>
Y Component from AUX	<p>Select this check box to replace the Y signal with the AUX input. This option essentially converts the lock-in to an A/D converter, providing a quick method for measuring an</p>

external signal. By default the check box is cleared.

Note that when the check box is selected, the channel will no longer function as a lock-in since its input value is overridden.

Outputs Tab

The Output tab options set the routing paths between the MAC III physical and internal connections.

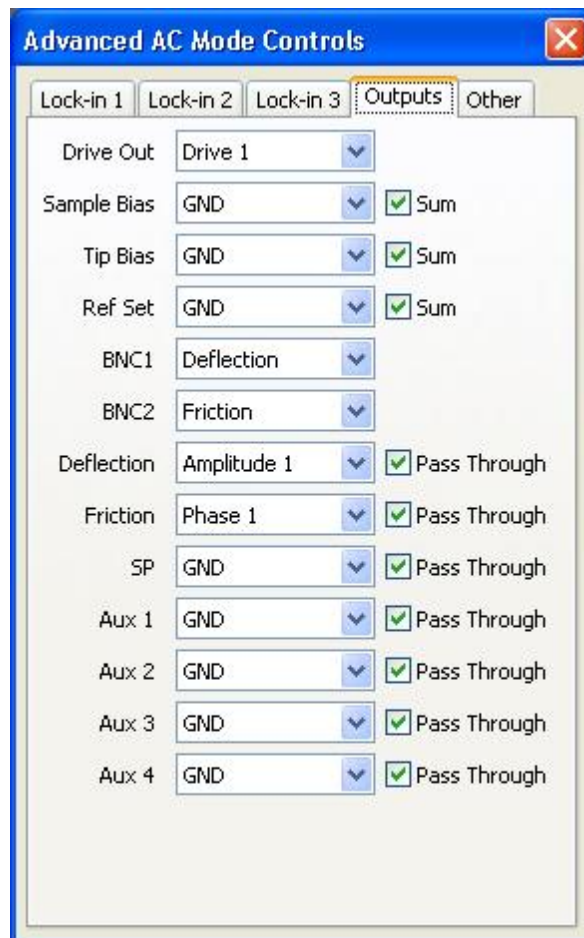


Figure 160 Advanced AC Mode Controls window: Outputs tab

Drive Out Routes the output from one of the three lock-in signals to the circuit controlling oscillation of the cantilever (either AAC or MAC, depending on your setup). Set this option to GND to turn off the output

from the MAC III. The default value is Drive 1 (the output from Lock-in 1).

Sample Bias

The Sample Bias is set in the Servo window and, by default, is sent from the AFM controller to the microscope. This option allows you to add the signal from one of the MAC III lock-ins to the Sample Bias, or to replace the Sample Bias completely. (Either Tip Bias or Sample Bias is selected in the Main tab).

First, select the Lock-in. Select the **Sum** check box to add the Lock-in signal to the Sample Bias; clear the check box to replace the Sample Bias with the lock-in signal.

Select the **Sum** check box plus **GND** to pass the Sample Bias from the AFM controller directly to the microscope. These are the default settings.

Tip Bias

The Tip Bias is also set under the Main tab of the Servo window and, by default, sent from the AFM controller to the microscope. The choice between Tip or Sample bias is made under the Advanced tab in the Servo window. This option allows you to add the signal from one of the MAC III lock-ins to the Tip Bias, or to replace the Tip Bias completely.

First, select the Lock-in. Select the **Sum** check box to add the Lock-in signal to the Tip Bias; clear the check box to replace the Tip Bias with the lock-in signal.

Select the **Sum** check box plus **GND** to pass the Tip Bias from the AFM controller directly to the microscope. These are the default settings.

Ref Set

Ref Set is the set point for the electrochemistry potentiostat.

First, select the Lock-in. Select the **Sum** check box to add the Lock-in signal to the Ref Set value; clear

the check box to replace the Ref Set value with the lock-in signal.

Select the **Sum** check box plus **GND** to pass the Ref Set value from the AFM controller directly to the microscope. These are the default settings.

BNC 1 and 2

The MAC III controller includes seven output channels: Deflection, Friction, SP and AUX 1-4. These output signals are routed to the AFM controller. They can also be duplicated at the two BNC connectors on the MAC III controller for additional routing flexibility.

By default, Deflection is routed to BNC1, and Friction is routed to BNC2. The actual signal sent to the BNC connectors is selected in the Output Channels, described next.

Outputs

The MAC III controller includes seven outputs (Deflection, Friction, SP, AUX 1-4) that are routed to the AFM controller for imaging.

Each output can carry one of thirteen signals: the Amplitude, Phase, X Component or Y Component of the three lock-in signals; or the output of the MAC III internal servo. Selecting GND for any output sets its output to 0.

By default, the Deflection output carries Amplitude 1 (the amplitude of Lock-in 1 output). The Friction output carries Phase 1. The remaining outputs are set to GND.

Select the **Pass Through** check box for each output to pass the signal directly from the microscope to the AFM controller without further contribution from the MAC III controller. By default, Pass Through is selected for each output.

NOTE

In KFM Mode, the SP (Surface Potential) channel is set to Servo Output and the **Pass Through** check box is cleared. The Servo Output is the DC bias produced by the servo to counteract the sample bias.

Other Tab

The Other tab includes additional parameters that control MAC III operation:

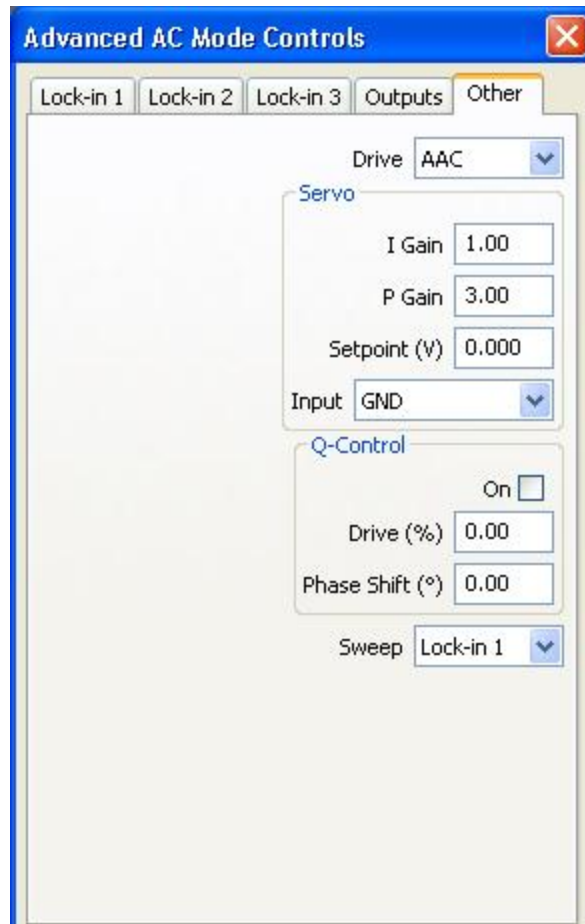


Figure 161 Advanced AC Mode window: Other tab

Drive	Selects the drive mechanism (AAC, standard MAC or Top MAC).
I Gain	The Integral Gain to the MAC III internal servo loop. The default value is 0.
P Gain	The Proportional Gain to the MAC III servo. The default value is 0.
Setpoint (V)	The voltage which the servo will try to maintain.
Input	Routes the Amplitude, Phase, X Component or Y Component from the three lock-in signals to the MAC III servo input. This is the signal that the servo

will maintain at the selected Setpoint. Selecting GND, the default setting, provides no signal to the servo.

Q Control On	By applying a phase-shifted version of the cantilever deflection signal on top of the drive signal, Q control can either increase or decrease the effective quality factor of the system. Select this On check box to enable the Q Control feedback loop. By default, Q Control is turned Off.
Drive (%)	Sets the amplitude of the Q Control phase-shifted signal, stated as a percentage (0-100 %) of the maximum available.
Phase Shift (°)	Shifts the Q Control feedback signal with respect to the input.
Sweep	Selects the lock-in for which the frequency will be swept on the AC Tune window. Only one lock-in can be swept at a time. Reverts to Lock-in 1 when AutoTune is selected in the AC Tune window. Use Manual Tune to sweep the other lock-ins.

Amplitude and Frequency Modulation Techniques

KFM can be performed in two main operations: In the first, the topography servo uses amplitude modulation (AM) to track the surface while the KFM servo uses amplitude modulation to track the surface potential. This first operation is referred to as AM mode.

In the second operation, the topography servo uses amplitude modulation to track the surface while the KFM servo uses frequency modulation (FM) to track the surface potential. The second operation is referred to as FM mode.

An optional signal access box is available for MAC III to allow custom multi-frequency experimentation like KFM FM mode. The box is shown in [Figure 151](#) on page 223 and described in [Table 4](#) on page 223. The auxiliary inputs and drive outputs are accessible through the MAC III Auxiliary Signal Access Box (ASAB). The software, which is flexible in routing signals back to the microscope controller, supports two servo systems related to these lock-in amplifiers.

The ASAB can be used in the development of novel capabilities for the examination of local mechanical and electromagnetic properties. This

section describes the hardware and software configurations of example experiments for EFM and KFM.

A block diagram for an example configuration of KFM FM mode using the Auxiliary Signal Access Box is shown in [Figure 162](#).

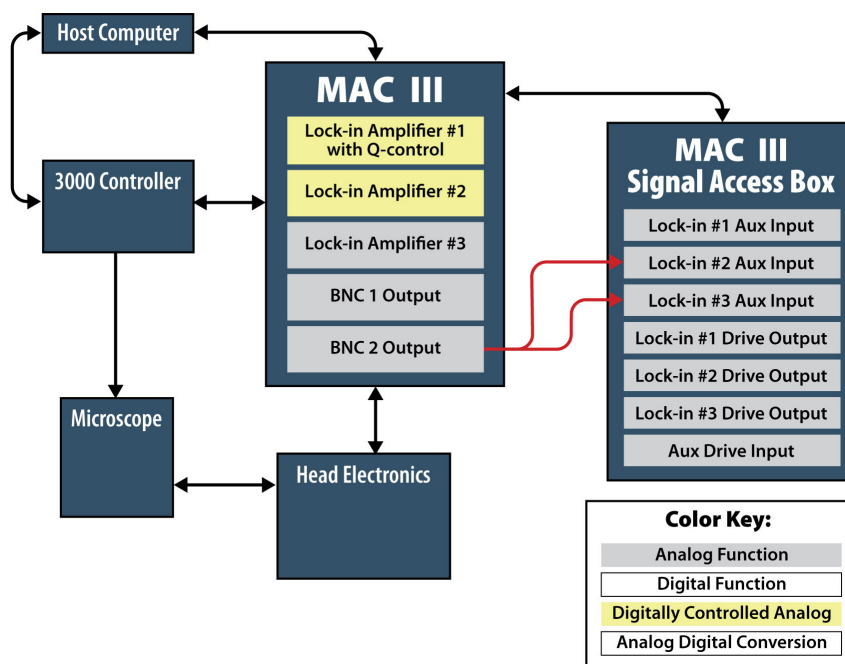


Figure 162 System block diagram for MAC III Signal Access Box configuration

In this KFM FM mode example, one lock-in amplifier is used for the amplitude modulation Z servo tracking the sample topography. The phase or alternately its X- or Y- vector components can be fed into a second lock-in amplifier to sense the small modulation in the effective free resonance of the cantilever caused by an electrostatic force gradient. The output of the second lock-in amplifier can be used as an input to the KFM servo used to null the electric field between the tip and sample. The third lock-in amplifier can be used for tuning the operational parameters, for recording the second harmonics of the electrostatic force gradient modulation which is proportional to dC/dZ , or for recording various signals (lateral response, torsional signal, harmonics, etc.) during measurements.

The routing of AUX input channels of the second lock-in amplifier and third lock-in amplifier to the BNC2 connector is used for most of the EFM and KFM applications. Typically, this configuration is used for the hardware setup (connections detailed in "[Connections for ASAB](#)" on

page 225) and the software is adjusted for different experimental conditions.

In KFM, the only difference from EFM is the use of an additional DC voltage. The following sections can be applied to either technique. When using EFM, the DC servo loop for KFM is not used.

NOTE

It is recommended that the sample be grounded. This can be done using a small piece of wire to connect the sample to the sample plate. The wire should go from the sample to the rectangular spring-loaded clamp (Figure 163) on the sample plate.

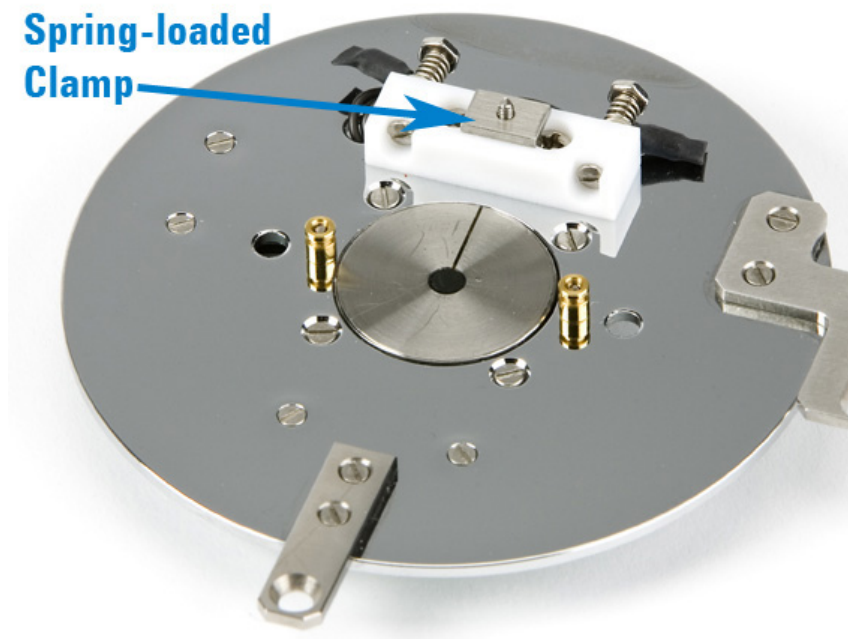


Figure 163 Connect a wire from the sample to the clamp

For tuning a KFM probe, Autotune can be performed from the KFM mode environment. For AM mode, it is generally beneficial to tune the cantilever below the resonant frequency. For FM mode, better results are achieved tuning the cantilever 200–300 Hz above the resonant

frequency. Once the Autotune has been completed, the sample can be approached to the tip with the approach button.

In the example below, the low frequency (first flexural resonance) was chosen, indicated by the red marker in [Figure 164](#).

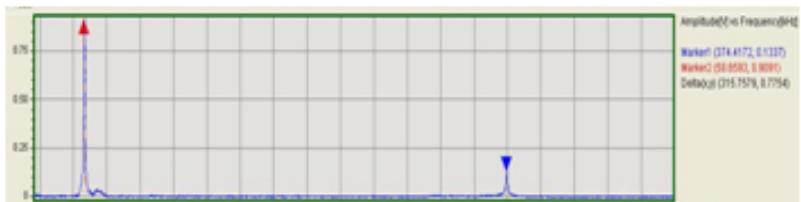


Figure 164 Frequency spectra for KFM probe

NOTE

A conducting probe is required for these applications.

NOTE

The following procedures are for PicoView 1.10.4 and higher.

Setting Parameters for AM Mode Operation

The KFM measurements can be performed in different ways. The same is true for EFM studies, which does not require using the electric servo loop needed in KFM.

KFM can be performed in two main operations: In the first, the topography servo uses amplitude modulation (AM) to track the surface while the KFM servo uses amplitude modulation to track the surface potential. This first operation is referred to as AM mode.

In the second operation, the topography servo uses amplitude modulation to track the surface while the KFM servo uses frequency modulation (FM) to track the surface potential. The second operation is referred to as FM mode and is described in more detail in "[Setting Parameters for FM Mode Operation](#)" on page 256.

Setting Parameters for Tracking Surface Topography

The Lock-in 1 parameters should be the same for all applications. These settings will yield measurements in the intermittent contact regime. This

procedure is for AM mode operation. Note the tip should *not* be engaged at this point.

- 1 Choose **Mode > KFM > AM**, then choose **Controls > AC Mode** to open the KFM Controls window.
- 2 Engage the tip and optimize the image.
- 3 Set parameters in the KFM tab as shown in [Figure 165](#) for initial settings.

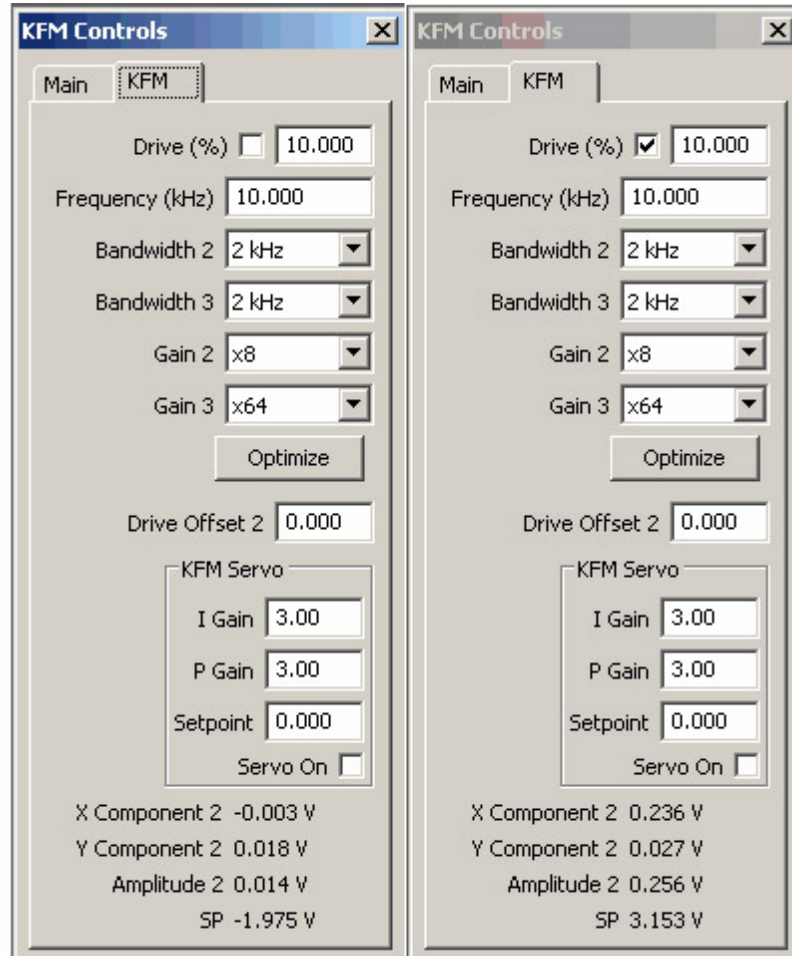


Figure 165 KFM tab initial settings (left) for AM mode operation. Settings on the right are after the Drive (%) check box has been selected and the Optimize button has been clicked.

- 4 Select the **Drive (%)** check box.

This will turn on the AC bias to the tip. If the Amplitude 2 parameter (in the lower portion of the KFM tab) is less than 0.100 V, increase the Drive (%) or Gain 2.

- 5 Click the **Optimize** button once.

This adjusts the phase shift so that the X component of Lock-in 2 is maximized positively and the Y component is minimized close to zero. These parameters should be verified in the lower portion of the KFM window.

- 6 Select the **Servo On** check box.

The SP parameter should stabilize to a single value. If it continues to fluctuate, try the following suggestions:

- a Clear the **Servo On** check box and set the drive offset to -3 V. Click the **Optimize** button and select the **Servo On** check box. Check for SP stability.
 - b If still unstable, try adjusting the KFM Servo Setpoint to -0.05 V or -0.1 V. If this stabilizes the SP, then the Setpoint needs further fine tuning. Instructions to do this are in [“Optimizing the KFM Servo Setpoint for AM Mode Imaging”](#) below.
 - c Clear the **Servo On** check box and increase the Drive (%). Click the **Optimize** button and select the **Servo On** check box. Check for SP stability.
- 7 In the Realtime Images window, add another imaging channel with the “+” button. At the top of the new window, select SP as the input.
- 8 If desired, another window can be opened and qualitative mapping of dC/dZ (handled by Lock-In 3) can be performed. Note that dC/dZ contrast is sample-dependent and is typically better in KFM FM mode.
- 9 Start scanning. Optimize the KFM controls to achieve the best SP image.
- a Adjust the KFM servo gains (I and P Gains) so that SP features are as sharp as possible. Avoid increasing them too much, though, as this can create noise in the SP and topography image.
 - b Adjust the bandwidth to achieve the highest SP resolution.
 - c It may be necessary to adjust topography I and P gains if the KFM servo affects topography imaging. Often lowering the Amplitude Setpoint helps to stabilize the topography.

Slower imaging speeds are recommended when using KFM.

Optimizing the KFM Servo Setpoint for AM Mode Imaging

When using AM mode, it is sometimes necessary to adjust the KFM Servo Setpoint to compensate for dependence on tip-sample separation. The fine tuning of the KFM Servo Setpoint parameter can be done by observing how the SP responds to changing tip height.

To check this, go to the Spectroscopy window, which should be set to Amplitude vs Distance, and open another channel to observe SP. Run an infinite number of sweeps over a very small distance range and observe how SP changes with distance. If SP is sloped, then adjust the KFM Servo Setpoint slowly until it is horizontal. [Figure 166](#) shows the SP sweep before and after the KFM Servo Setpoint has been adjusted. Note the difference in the vertical scale between before and after.

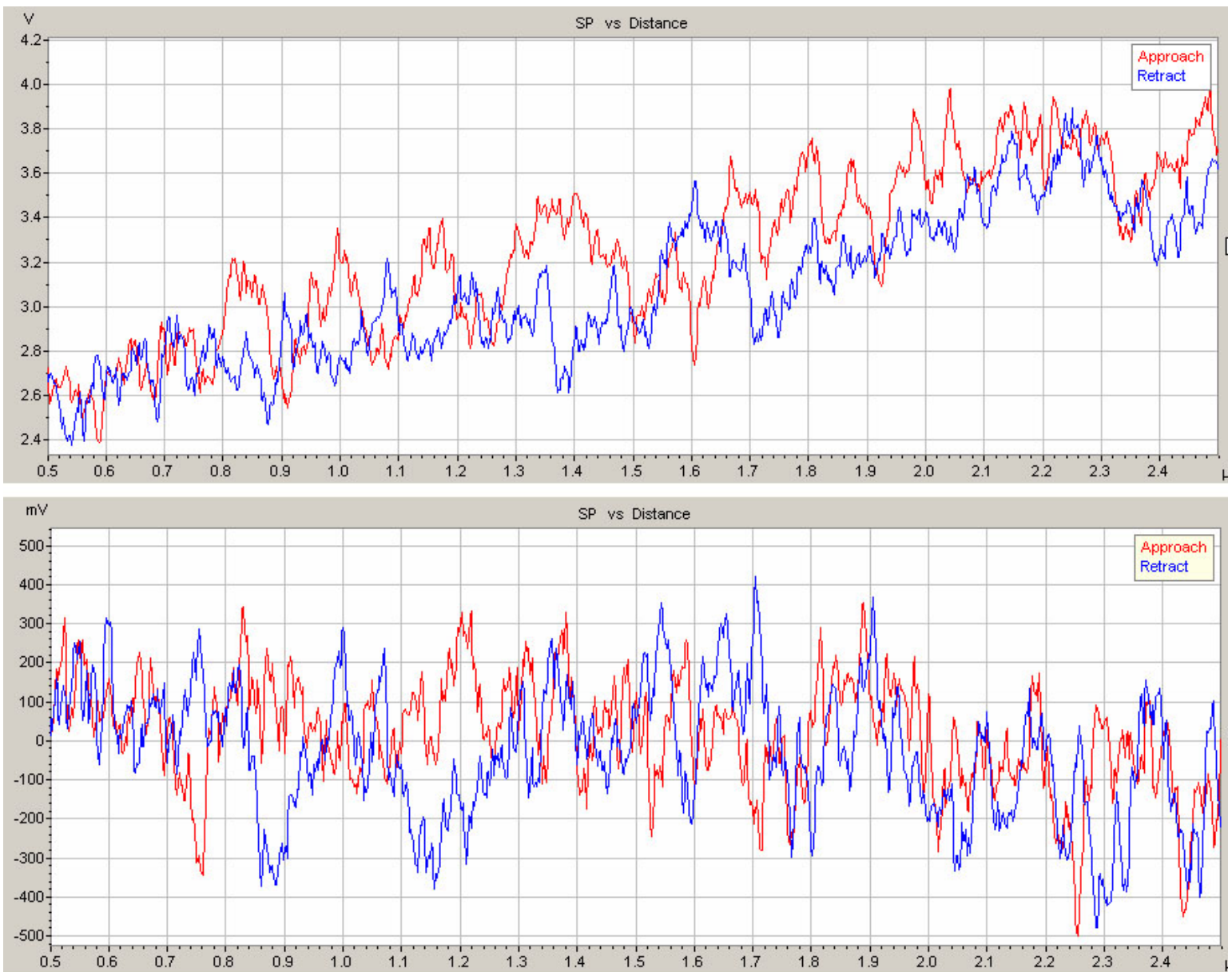


Figure 166 SP vs Distance before (top) and after (bottom) the KFM Servo Setpoint adjustment

Setting Parameters for FM Mode Operation

KFM imaging in FM mode requires the use of the auxiliary signal access box (ASAB). Connect the ASAB to the controller as shown in [Figure 152](#) on page 225. The ASAB is connected to the auxiliary serial

port on the back of the MAC III. A BNC cable should connect the BNC2 output on the MAC III controller with the Lock-in 2 input on the ASAB. If dC/dZ imaging is desired, the BNC2 output should also go to the input of Lock-in 3.

Choose **Mode > KFM > FM**, then choose **Controls > AC Mode** to open the KFM Controls window. Once the tip is engaged on the surface of the sample and topography imaging is optimized, the KFM Controls window can be used to optimize the KFM parameters.

The default parameters for FM are shown in [Figure 167](#).

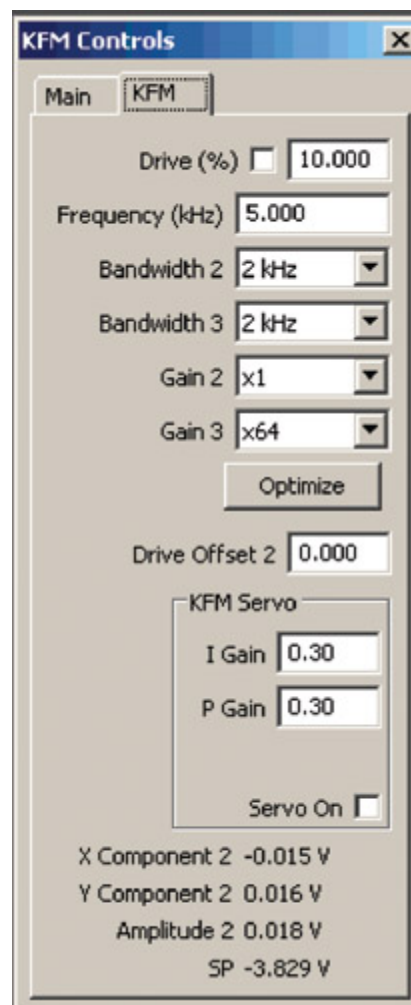


Figure 167 KFM tab initial settings for FM mode operation

Note there is no KFM Servo Setpoint parameter in FM mode. The instructions for preparing to image are very similar to the instructions for AM mode. The steps to use the control window for KFM imaging are below.

- 1 Select the **Drive (%)** check box.

This will turn on the AC bias to the tip. If the Amplitude 2 parameter (in the lower portion of the KFM tab) is less than 0.04 V - 0.05 V, increase the Drive (%) or Gain 2.

- 2 Click the **Optimize** button once.

This adjusts the phase shift so that the X component of Lock-in 2 is maximized positively and the Y component is minimized close to zero. These parameters should be verified in the lower portion of the KFM window.

- 3 Select the **Servo On** check box.

The SP parameter should stabilize to a single value. If it continues to fluctuate, try the following suggestions:

- a Clear the **Servo On** check box and set the drive offset to -3 V. Click the **Optimize** button and select the **Servo On** check box. Check for SP stability.
 - b Clear the **Servo On** check box and increase the Drive (%). Click the **Optimize** button and select the **Servo On** check box. Check for SP stability.
- 4 In the Realtime Images window, add another imaging channel with the “+” button. At the top of the window, select SP as the input.
 - 5 If desired, another window can be opened and qualitative mapping of dC/dZ (handled by Lock-In 3) can be performed. Note that dC/dZ contrast is sample-dependent.
 - 6 Flattening for SP and dC/dZ images should be set to **None**.
 - 7 Start scanning. Optimize the KFM controls to achieve the best SP image.
 - a Adjust the KFM servo gains (I and P Gains) so that SP features are as sharp as possible. Avoid increasing them too much, though, as this can create noise in the SP and topography image.
 - b Adjust the bandwidth to achieve the highest SP resolution.
 - c It may be necessary to adjust topography I and P gains if the KFM servo affects topography imaging. Often lowering the Amplitude Setpoint helps to stabilize the topography.

Slower imaging speeds are recommended when using KFM.

Piezoresponse Force Microscopy

Piezoresponse Force Microscopy (PFM) is a scanning probe microscopy technique based on the reverse piezoelectric effect, where a piezoelectric material expands or contracts upon applying to it an electric field. PFM mode has been shown to delineate regions of different piezoresponse with subnanometer lateral resolution.

PFM mode is a type of contact mode and requires a SPM probe that is coated with a conductive material. An AC modulation with an optional DC offset bias is applied to the tip, which is in contact with the sample surface, and the piezoresponse of the sample is measured from the deflection of the cantilever.

PFM Spectroscopy is a non-imaging technique that compliments PFM imaging. In PFM Spectroscopy, the piezoresponse of a given location on the sample can be mapped versus, for example, the DC bias or the frequency of the AC signal applied to the sample via the tip. This technique can also be used to study hysteretic characteristics of the piezoresponse.

12 Liquid Cell

Liquid Cell with Standard Sample Plate 261

Liquid Cell with MAC Mode 263

Flow-Through Liquid Cell 263

The liquid cell enables *in-situ* AFM or STM imaging for better control under realistic environments. The cell is made from chemical-resistant polycarbonate and can be used with a wide variety of liquids. The cell can be used in conjunction with a standard, MAC Mode or temperature control sample plates.

A flow-through version of the liquid cell is also available with 0.9 mm holes included for tubing.

Eight-degree nose assemblies are recommended for imaging in liquid because the smaller angle takes into account the different angle the laser makes as it goes in and out of the fluid, compared to operation in air. A Top MAC nose assembly is available with an eight-degree nose assembly option.

CAUTION

Some nose assemblies, such as the two-piece nose assemblies and CSAFM nose assembly, are not sealed and should never be used for imaging in liquid. Be sure to use only approved nose assemblies for imaging with the liquid cell.

Figure 168 shows the components of the liquid cell: two retaining clips, an O-ring gasket and the liquid cell plate. **Figure 169** shows the components as assembled on a standard sample plate. Note that, when assembled, the sample itself comprises the bottom of the liquid

container; therefore, the sample must be large enough for the O-ring to seat.

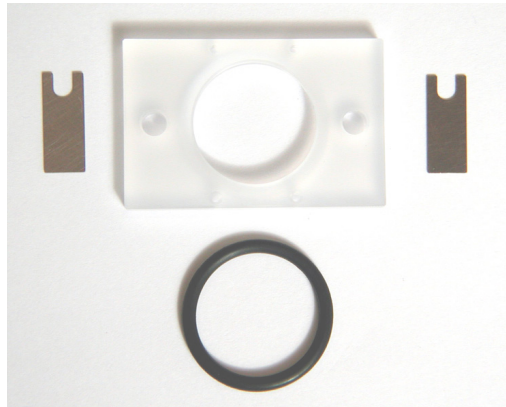


Figure 168 Liquid cell components

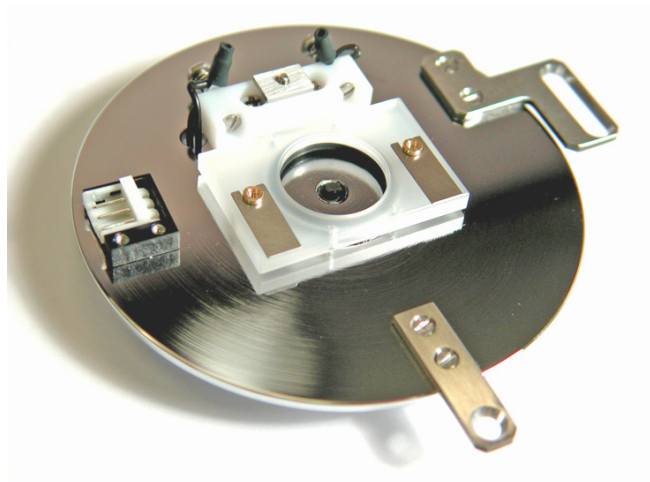


Figure 169 Liquid cell mounted on standard sample plate

Liquid Cell with Standard Sample Plate

One challenge with using the liquid cell is to locate the region of interest through the liquid. It is typically easier to first locate the dry sample and then to add the liquid, as follows:

- 1 Prepare and place the sample on the sample plate.
- 2 Place the sample plate on the microscope.
- 3 Using the **Close** switch on the Head Electronics Box, and watching the video system, roughly approach the sample such that the tip and

sample are almost touching (i.e., both the tip and sample are close to focus).

- 4 In PicoView, click the **Approach** button to place the tip in contact with the surface.
- 5 Use the video system to locate the region of interest.

CAUTION

Be extremely careful when moving the scanner while using the liquid cell. Clearance is limited, and contact between the scanner and cell will damage the scanner.

- 6 Now, in PicoView click the **Withdraw** button to take the tip out of contact with the surface.
- 7 Use the motor controls to move the scanner several millimeters from the sample, providing enough clearance to safely remove the sample plate. **Be sure to note the distance that the scanner is moved**—you will need it to accurately re-engage the tip and sample shortly.
- 8 Remove the sample plate from microscope.
- 9 Place the O-ring on the sample plate so that it encompasses the sample.
- 10 Place the liquid cell plate over the O-ring and sample, such that it aligns with the sample plate's spring-loaded pins.
- 11 Push one of the pins up through the liquid cell plate and slide one of the retaining clips into the groove in the pin. Repeat for the other pin. The liquid cell is now firmly attached and sealed to the sample plate.

NOTE

Make sure that the liquid cell plate sits flat against the sample plate to create a good seal and prevent leakage. If necessary, use a flat head screwdriver to adjust the tension on the two retaining pins.

- 12 Add the appropriate liquid to the cell. Use enough liquid to submerge the sample but not so much that the liquid rises to the top of the cell. This will prevent spillage as the scanner moves into the cell.
- 13 Replace the sample plate on the microscope.
- 14 Move the scanner down the same distance it was moved up in [step 6](#), such that the tip is now just above the sample surface. You will need to adjust the photodetector position due to the change of the laser location caused by the laser now going through liquid.
- 15 If necessary, readjust the photodetector position to account for the change of laser location caused by the liquid.
- 16 Approach and contact the sample.

- 17 Because of the large movements involved in placing and aligning the liquid cell, you will likely need to adjust both the detector and the scanner position before imaging.

Liquid Cell with MAC Mode

The procedure for setting up the cell for MAC Mode imaging is similar to that described above. However, the MAC mode sample plate contains a ferrite core that can react when placed in solution or in contact with the sample. Therefore, a cover slip should be placed over the core, and the sample placed on the cover slip, to ensure that the core does not contact the sample or liquid.

Flow-Through Liquid Cell

A liquid cell is also available with connections allowing liquid to flow continuously through the cell. The connections should be made with 1 mm OD Teflon tubing, cut at a sharp angle for insertion into the cell.

13 Temperature Control

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Several temperature control sample plates are available for use with the Keysight 5500 SPM. With temperature control, studies can be done while maintaining physiological temperature, for melting experiments, etc.

Cantilevers for Temperature Controlled Imaging

Uncoated silicon cantilevers are recommended for imaging under temperature control. Cantilevers that are coated on one side will bend

due to the difference in thermal expansion of the coated and uncoated sides. The bending may adversely affect imaging.

High Temperature Sample Plates

Two high temperature sample plates are available. The standard hot sample plate (Figure 170) provides a temperature range from ambient to 250 °C.

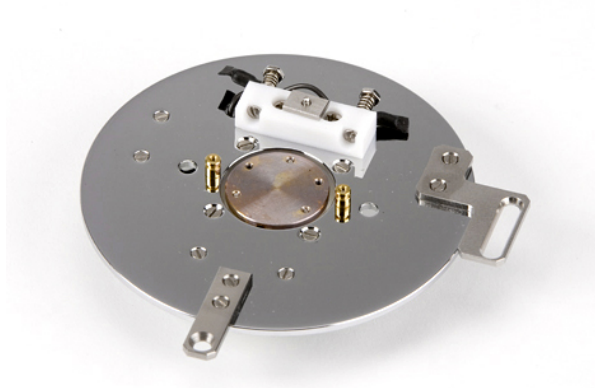


Figure 170 Standard hot sample plate

The Hot MAC sample plate (Figure 171) provides temperatures from ambient to 110 °C and enables imaging in MAC Mode.

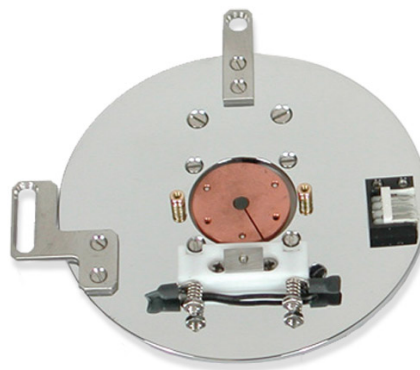


Figure 171 Hot MAC sample plate

The Lakeshore 332 Temperature Controller (Figure 172) drives the high temperature plates. Note the temperature sensor type in the Lakeshore Control Setup menu should be set to **100 Ω Plat/500**.



Figure 172 Lakeshore temperature controller

CAUTION

The ramping rate should be kept below 10 degrees per minute to avoid damaging the plate.

Connections

Figure 173 shows the three cables included with both high temperature sample plates:

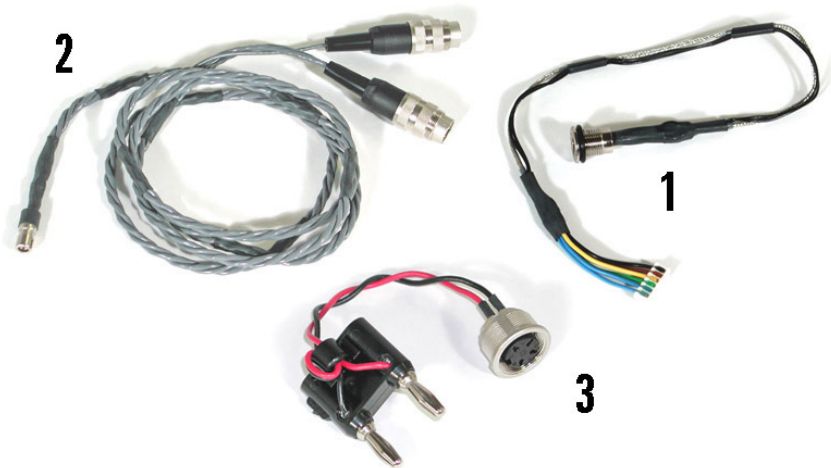


Figure 173 High temperature sample plate cables

The Hot MAC sample plate also includes a Y connector for the MAC cable (Figure 174).



Figure 174 Hot MAC sample stage Y cable.

Figure 175 shows the required wiring for the hot sample plate.

Figure 176 shows the wiring for the hot MAC sample plate.

The connection at the end of Cable 1 enables wiring to the temperature stages through a port in the environmental chamber. Fold the straight

connector parallel to the cable and pass it through a port in the chamber. Then screw the round connector into the port to make a tight seal.

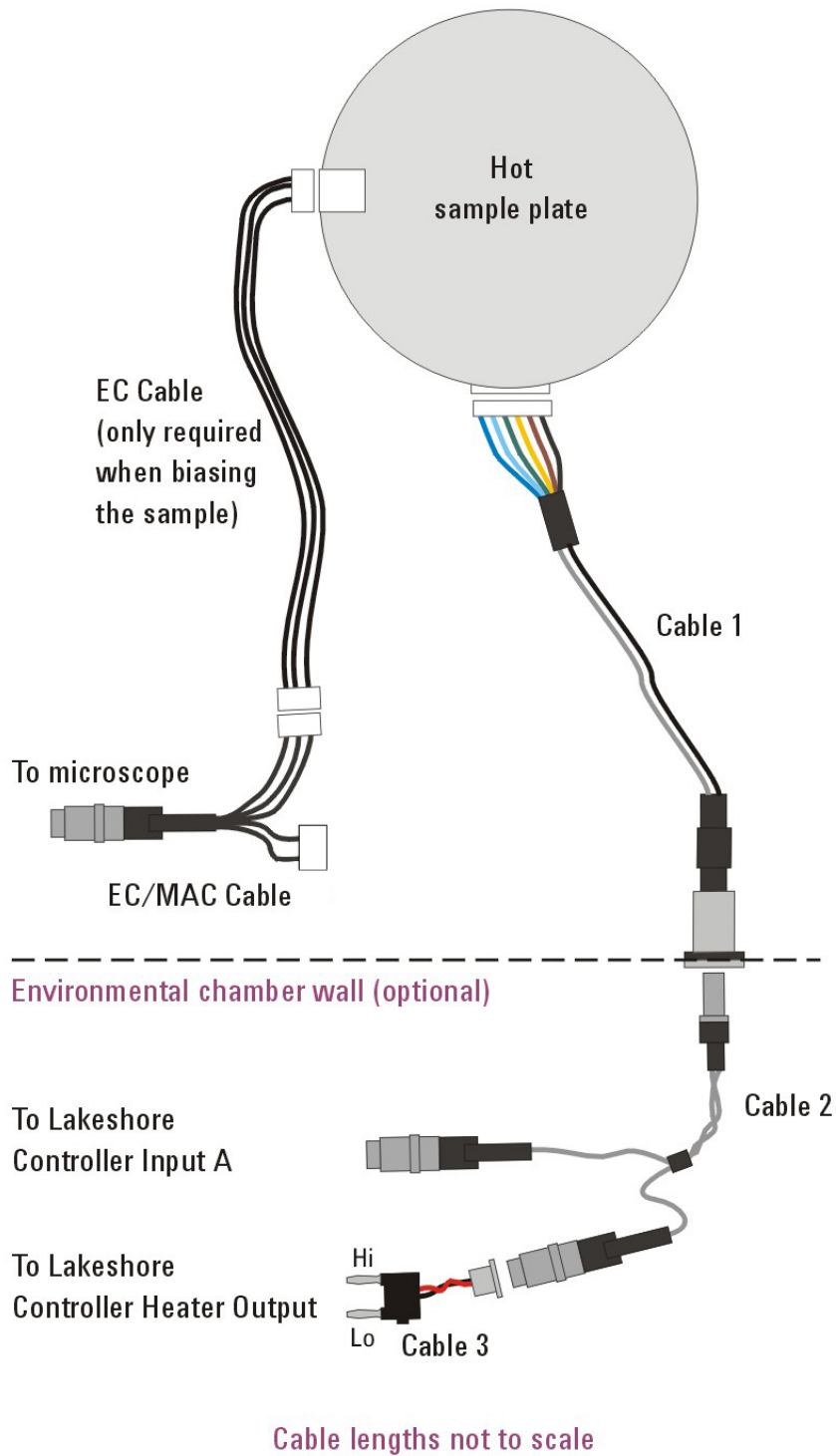


Figure 175 Hot sample plate wiring diagram

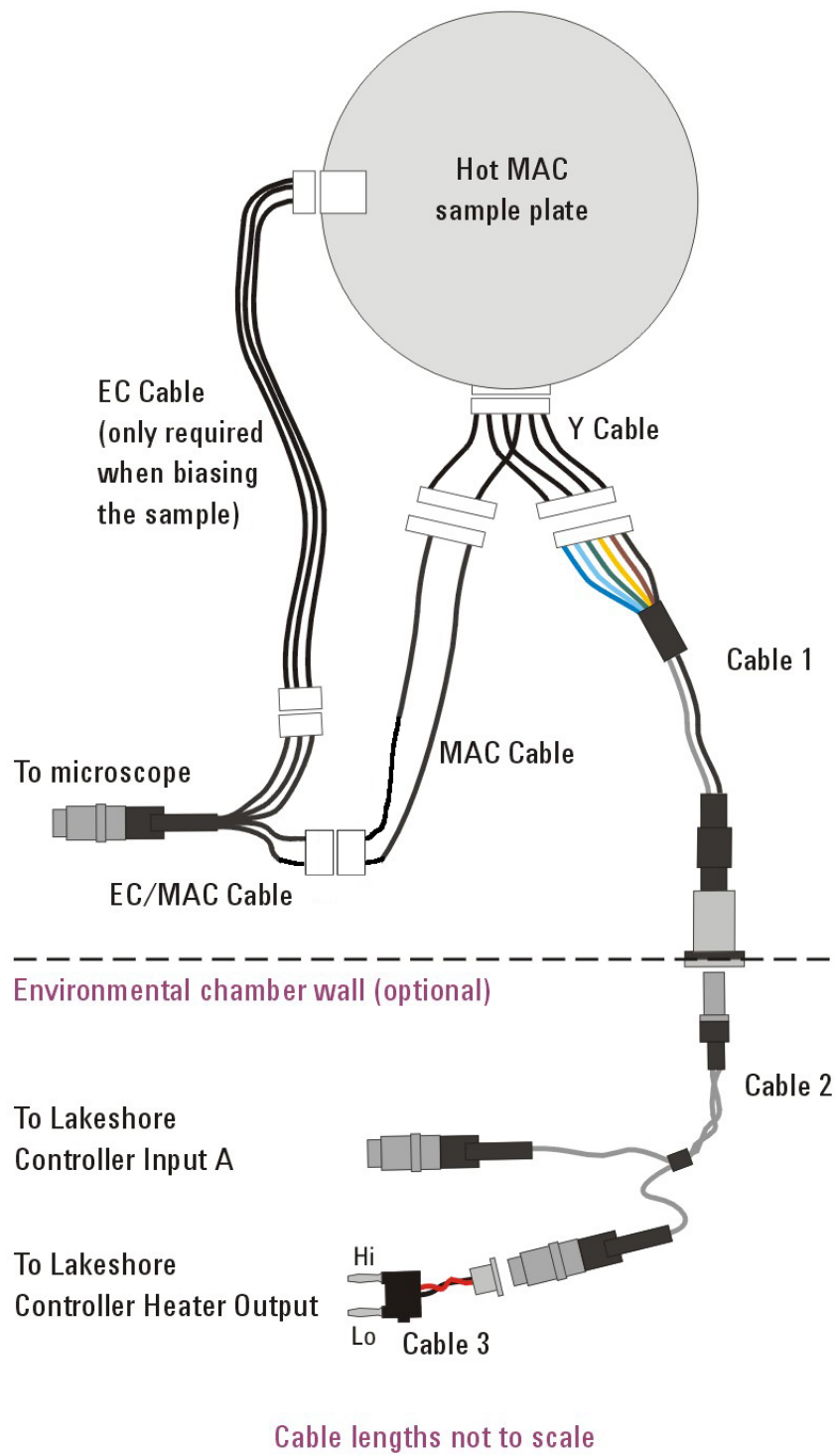


Figure 176 Hot MAC sample plate wiring diagram

Note that Cable 3 includes a tab on the black-wire side of the connector (Figure 177). The tab must face the Lo jack on the controller.



Figure 177 Tab side of Cable 3 must be inserted into the Lo jack.

Imaging

- 1 Set up the microscope for typical operation. As mentioned, uncoated silicon probes are highly recommended.
- 2 Mount the sample on the sample plate. Do not use double-sided tape to mount the sample because the glue may soften or melt, causing large sample drift.
- 3 Turn on the Lakeshore temperature controller.
- 4 On the Lakeshore controller's front panel, press the **Heater Off** button.
- 5 Press **Auto Tune**, then press the **Up** or **Down** arrow buttons until the display read **Tune:Manual**.
- 6 Set the Proportional, Integral and Differential gains. Typical values are **20, 20** and **100** respectively.
- 7 Press **Setpoint** and enter a value slightly lower than room temperature (23 °C). Wait for the setpoint value to stabilize.

NOTE

When turned on, the Lakeshore controller will attempt to adjust the plate temperature to the last selected temperature, as quickly as possible. Depending on the sample, and the last temperature setting, this can be detrimental to the plate and/or sample. Beginning with a setpoint slightly below ambient avoids this problem.

- 8 Set the Ramp Rate to no more than **10 degrees per minute** (5 degrees/minute is a typical ramp rate).

- 9 Press the **Heater Range** button and select **Low**, **Medium** or **High**.

CAUTION

Do NOT use the **High** setting with a Peltier (cooling) plate (see below).

- 10 Press the **Setpoint** button and enter the desired final temperature.
- 11 Allow the temperature to stabilize.
- 12 Initiate an approach.
- 13 Image as usual.

Imaging during temperature ramp is possible provided care is taken to compensate for sample thermal expansion. Monitor the Z-piezo position as the temperature increases to determine if/when it goes out of range.

Peltier (Cold MAC) Sample Plate

The Peltier Cold MAC sample plate lets you image in contact, AAC, MAC or STM Modes at controlled temperatures below or near ambient temperature (Figure 178). The 1X Peltier plate provides a temperature range of -5 to 40 °C.

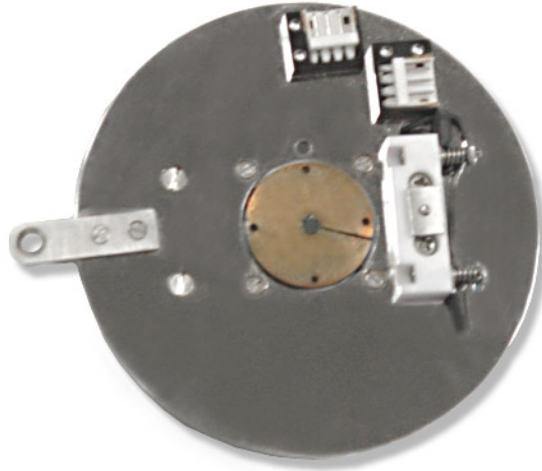


Figure 178 Peltier (Cold MAC) sample plate.

The Lakeshore 332 Temperature Controller (Figure 172 on page 266) is also used with the Peltier plate.

The current booster (Figure 179) is used to drive the Peltier sample plate temperature. The booster includes a safety device that shuts off the power if the reverse side of the Peltier becomes excessively hot.



Figure 179 Current booster

Connections

Figure 180 shows the three cables included with the Peltier sample plate:

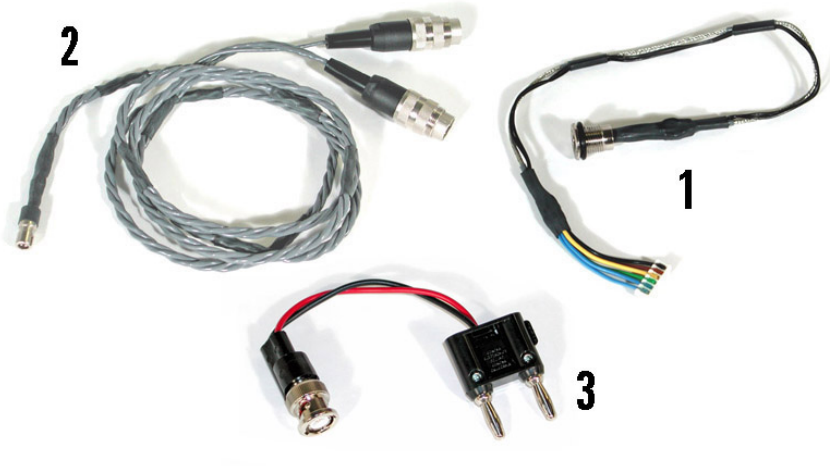


Figure 180 Peltier sample plate cables

The Peltier sample plate also connects to MAC cables for use with MAC Mode (Figure 181).

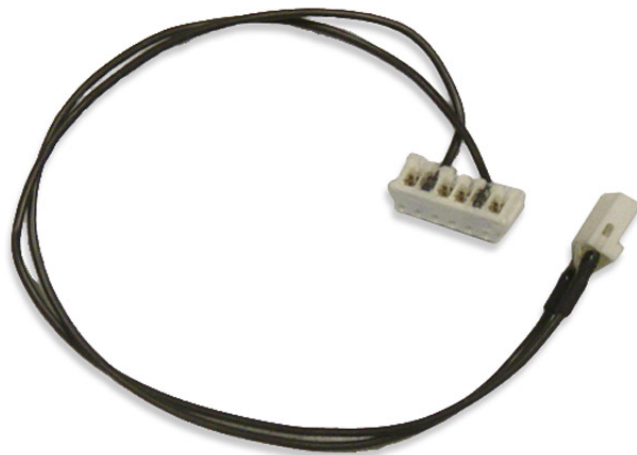


Figure 181 MAC cable for Peltier sample plate

Figure 182 shows the required wiring for the Peltier sample plate. MAC Mode is optional and the figure below shows the setup with the MAC

Mode option. The connection at the end of Cable 1 enables wiring to the temperature stages through a port in the environmental chamber.

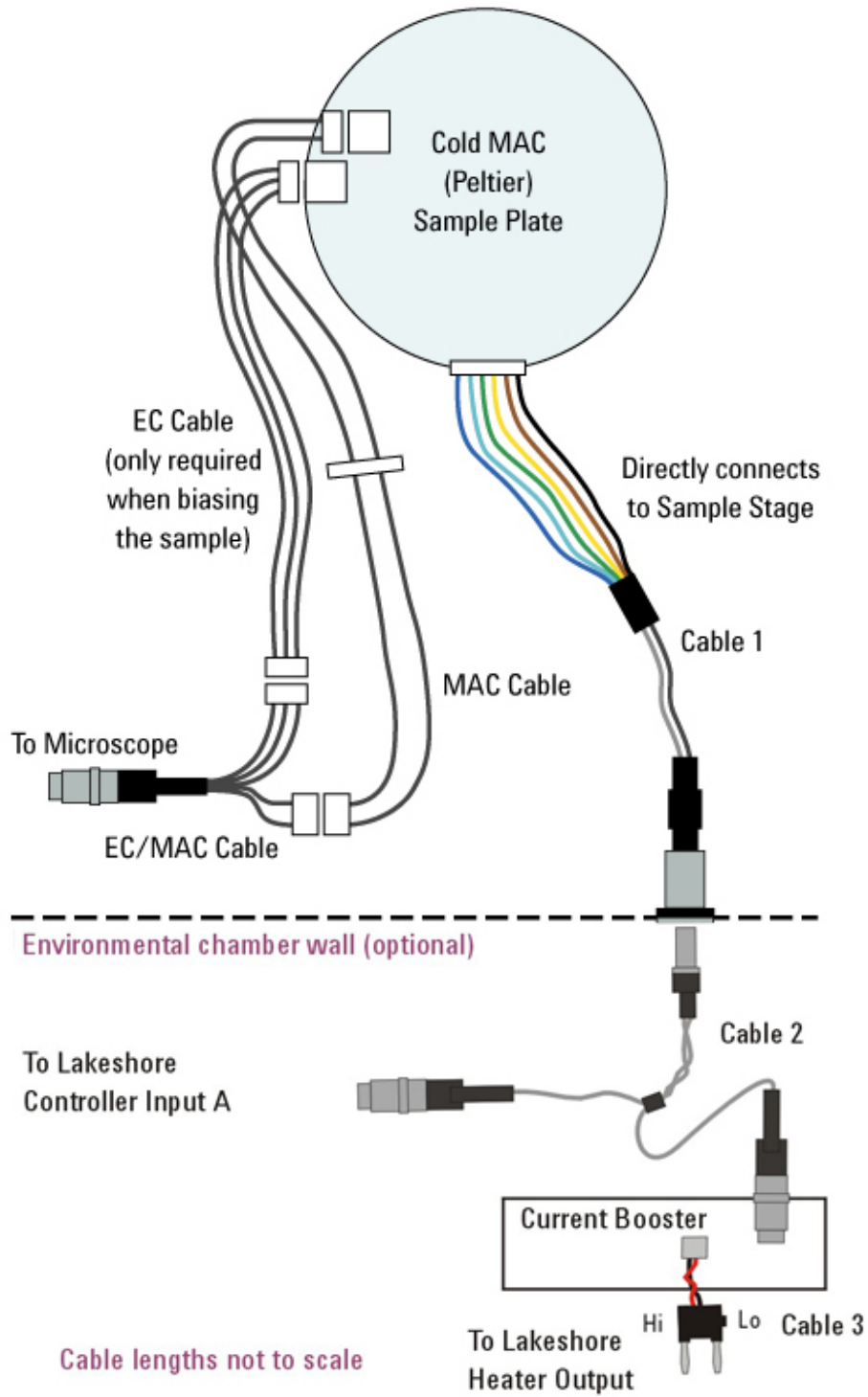


Figure 182 Peltier (Cold MAC) sample plate wiring diagram.

Note that Cable 3 includes a tab on the black-wire side of the connector (Figure 183). The tab must face the Lo jack on the controller.



Figure 183 Tab side of Cable 3 must be inserted into the Lo jack

Water Cooling

When a sample is cooled using the Peltier sample plate the opposite side of the Peltier device becomes hot. The hot side is water cooled to decrease the minimum sample temperature, reduce power requirements and prevent overheating. Inlets are provided on the underside of the Peltier sample plate to connect water cooling tubing.

A gravity-fed water-cooling system (Figure 184) is preferred over mechanical pumping because it reduces the potential for vibration that can affect imaging. Two reservoirs are provided with the Peltier sample plate, one to be used as a source and the other as a receptacle to store water for recycling. A height difference of three feet between the source and receptacle gives a minimum temperature of $-25\text{ }^{\circ}\text{C}$ or approximately $50\text{ }^{\circ}\text{C}$ below room temperature. Ice can be added to the reservoir to increase efficiency for a minimum temperature of $-30\text{ }^{\circ}\text{C}$.

CAUTION

Connect the water-cooling system and test for leaks *before* making any electronic connections or turning on any components.

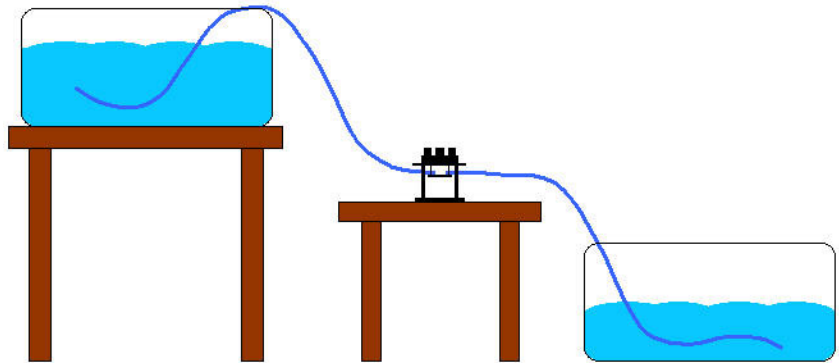


Figure 184 Gravity-fed water cooling system for Peltier sample plate.

Imaging

- 1 Set up the microscope for typical operation. As mentioned above, uncoated silicon probes are highly recommended.
- 2 Mount the sample on the sample plate.
- 3 Set the **Range** control on the current booster to its minimum setting (fully counterclockwise).
- 4 Set the current booster to **1X**.
- 5 Turn on the Lakeshore controller and current booster.
- 6 On the Lakeshore controller's front panel, press **Auto Tune**, then press the **Up** or **Down** arrow buttons until the display read **Tune:Manual**. Press **Enter**.
- 7 Set the Proportional, Integral and Differential gains. Typical values are **12**, **12** and **5** respectively.
- 8 Press **Setpoint** and enter a value slightly lower than room temperature 23 °C. Wait for the setpoint temperature to stabilize.

NOTE

The Lakeshore controller will attempt to adjust the temperature to the last selected temperature as quickly as possible. Depending on the sample, and the last temperature setting, this can be detrimental to the plate and/or sample. Setting the temperature to ambient avoids this problem.

- 9 Set the Ramp Rate to no more than **10 degrees per minute** (5 degrees/minute is a typical ramp rate).
- 10 Turn the **Range** control on the current booster to maximum (fully clockwise).

- 11 On the Lakeshore controller press **Heater Range** and select **Low**.
- 12 Press the **Setpoint** button and enter the desired final temperature.
- 13 Allow the temperature to stabilize.
- 14 Initiate an approach.

Tips for Temperature Controlled Imaging

- Make sure there is good thermal contact between the sample and the sample plate. If possible, mount the sample using the liquid cell even for ambient imaging.
- Double-sided tape reduces thermal conductivity as well as introducing sample drift. Therefore it should not be used to mount samples for temperature controlled experiments.
- It is possible to ramp the temperature while imaging. Use slow ramps, typically less than 1 °C per minute.
- Every sample will react differently to temperature control. A thin piece of graphite is a good test sample to use while setting up the temperature control system.
- Temperature fluctuations due to excessive gains will cause the surface to appear wavy. Reduce gains on the Lakeshore controller to reduce this waviness.
- Imaging in the environmental chamber is recommended as it will help keep the temperature stable. Purging the environmental cell with a dry gas such as nitrogen will help control the sample environment if condensation from cooling to below the dew point becomes an issue.

14 Thermal K

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Spring Constant Calibration	282

It is sometimes necessary to know the spring constant of an AFM probe. This chapter reviews the calibration method using Thermal K to determine spring constants.

Atomic Force Microscopy (AFM) is a powerful technique that can be used to quantify the elastic properties of materials or to measure single molecule unfolding or unbinding interactions on the picoNewton scale. AFM probes are composed of flexible, triangular or rectangular cantilevers with a sharp tip near the end of the cantilever. They can be manufactured from a variety of materials, but most AFM probes are made from silicon and/or silicon nitride (Si_3N_4) wafers using semiconductor-based etching processes.

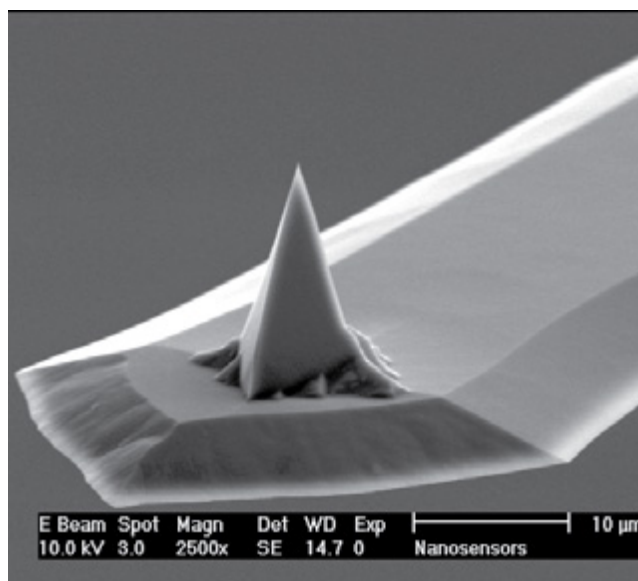


Figure 185 Scanning electron microscopy image of an AFM probe

An AFM probe's sensitivity, or spring constant (κ), is the force required to bend the cantilever per unit distance (usually reported in Newtons/meter; N/m). It is an important factor in AFM probe behavior and performance for imaging applications and an essential parameter when attempting to quantify intra- or inter-molecular interactions or the compliance of materials with the AFM.

Nominal spring constant values are commonly reported by AFM probe suppliers. However, because of manufacturing variability, the spring constants of individual cantilevers can vary greatly; even between probes in the same batch or from the same wafer. Consequently, in order to accurately quantify material compliance or molecular interactions with the AFM, the spring constant for each probe should be determined by empirical or semi-empirical methods.

Thermal K Calibration

Thermal K is an option for the Keysight AFM that allows the user to quickly and easily calculate the spring constant of an AFM probe. The Keysight Thermal K method calculates AFM probe spring constants by describing the motion of the cantilever as a harmonic oscillator through the use of the equipartition theorem from fundamental thermodynamic theory.

The theory states that the kinetic energy stored in a system at a momentum coordinate, which is the deflection of a cantilever from its equilibrium position, is equal to one half of the thermal energy of the system.* The deflection value from the resting state of the cantilever is considered to be small for AFM probes, and it can be assumed to be linearly related to the force required to deflect the cantilever according to Hooke's Law:

$$F = -\kappa \cdot x$$

For an ideal spring with a spring constant value of κ , the thermal noise according to its position allows the spring constant to be determined. In principle, simply measuring the deflection value of the cantilever and temperature would permit its spring constant to be calculated. However, an AFM cantilever is not an ideal Hookean spring, so its potential energy can not accurately be described in such a simple manner. Consequently, the area of the resonant peak in the Power Spectrum Density (PSD) spectra of the vibrational noise permits a more accurate determination of κ .

Thermal K Setup

The Thermal K method requires installation of an additional data acquisition card. To enter the acquisition card address, open the Setup section of the Controls drop down menu and select Components. Enter

*S.M. Cook, T.E. Schaffer, K.M. Chynoweth, M. Wigton, R.W. Simmonds, and K.M. Lang, *Nanotechnology* Volume 17, 2135 (2006).

the address in the Acquisition Card Address field and select the Acquisition Card (Thermal K) check box to activate the card.

Before the spring constant can be computed, the **Deflection Sensitivity** has to be known. This number can be entered directly in the Spectroscopy Advanced tab, or it can be computed as described in the [Spring Constant Calibration](#) section below.


A rigid sample like mica will also be needed to obtain a good force curve.

NOTE

In order to obtain accurate spring constant values with Thermal K, the Z closed loop scanner is recommended. Also, in order to maintain the integrity of the tip of the AFM probe, it is often preferable to perform the calibration after all Force vs Distance sweeps have been performed. PicoImage is required for post-processing. The **Cantilever Settings** button in PicoImage allows the user to set (or calculate) the Sensitivity, enter a spring constant after the fact, and then display the plot with V, nm or nN vertical scale units.

Spring Constant Calibration

In the PicoView software main window:

- 1 Choose **Mode >Contact**.
- 2 Choose **Controls >Spectroscopy** to open the Spectroscopy window.
- 3 In the Spectroscopy window, confirm **Force vs Distance** for the plot type ([Figure 186](#)).
- 4 In the Spectroscopy Basic tab, click on the 1 button  to do a single sweep.

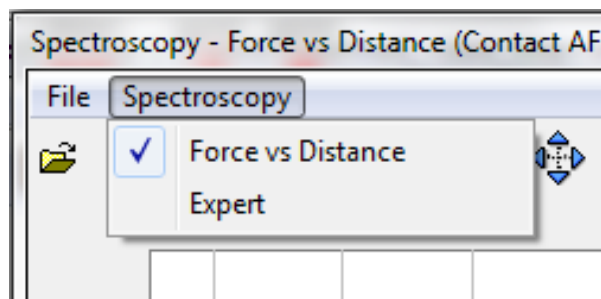


Figure 186 Spectroscopy window showing Force vs Distance selected

- 5 Obtain a force curve using a rigid substrate like freshly cleaved mica.
- 6 In the plot window, right-click to **Add Ruler to Plot** on a steep portion of the deflection curve. Click on the ruler line to move the second marker closer to the jump-to-contact point as shown below.

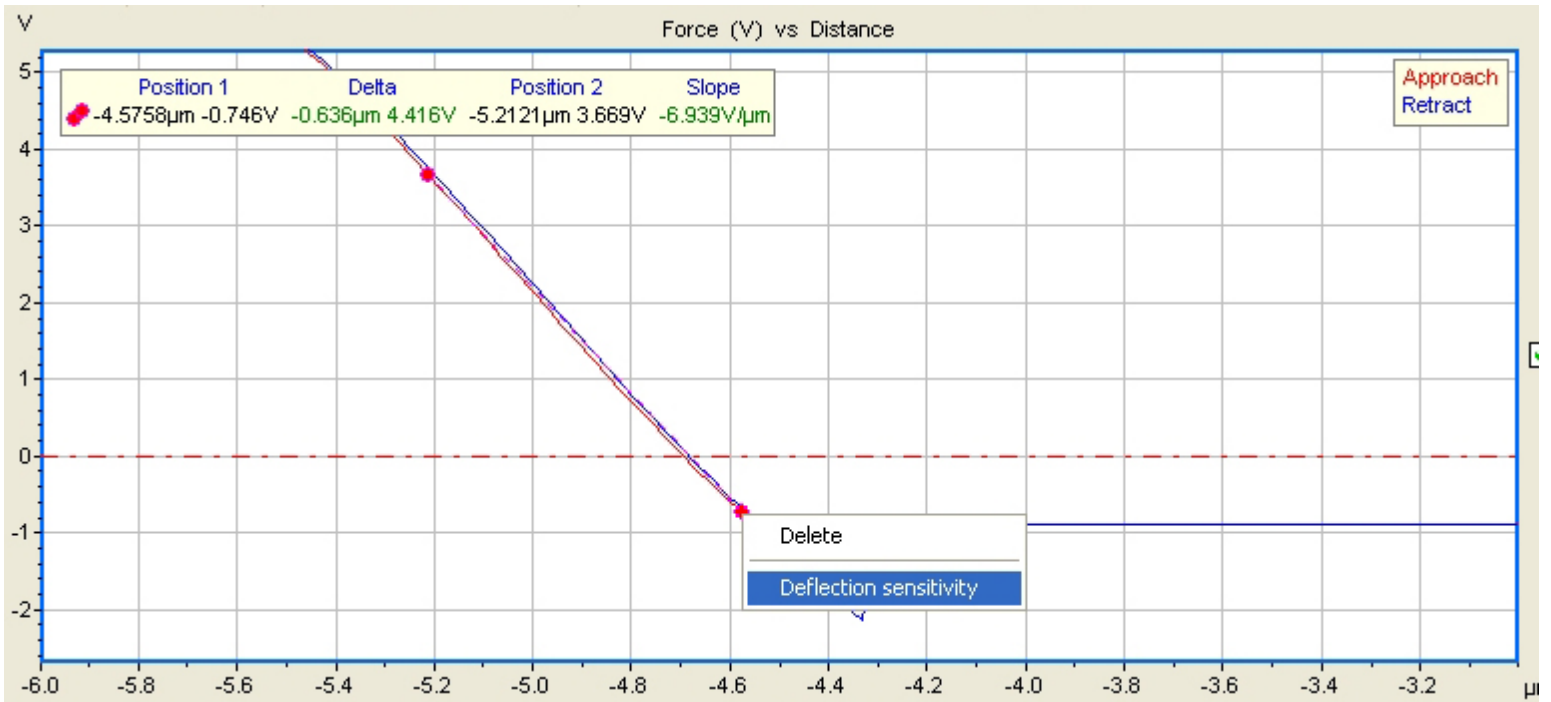


Figure 187 Force curve (Deflection vs distance) with ruler markers

- 7 Right-click on the ruler after the end points are set and select **Deflection Sensitivity** (Figure 188) to update the value in the Advanced tab.

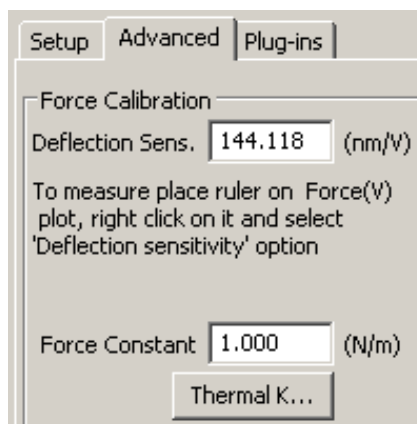
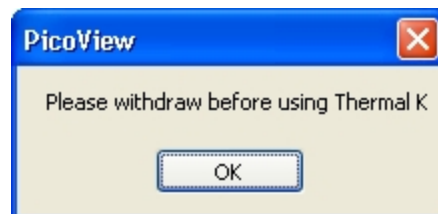


Figure 188 Advanced tab in the Spectroscopy window

- 8 Click the **Thermal K...** button in the Advanced tab of the Spectroscopy window.

A message window will open as a reminder to withdraw the tip.



- 9 Click **OK**.
- 10 Click on the Withdraw button at the top of the main window to withdraw the tip.

This will open the Thermal K plot window, similar to that shown below.

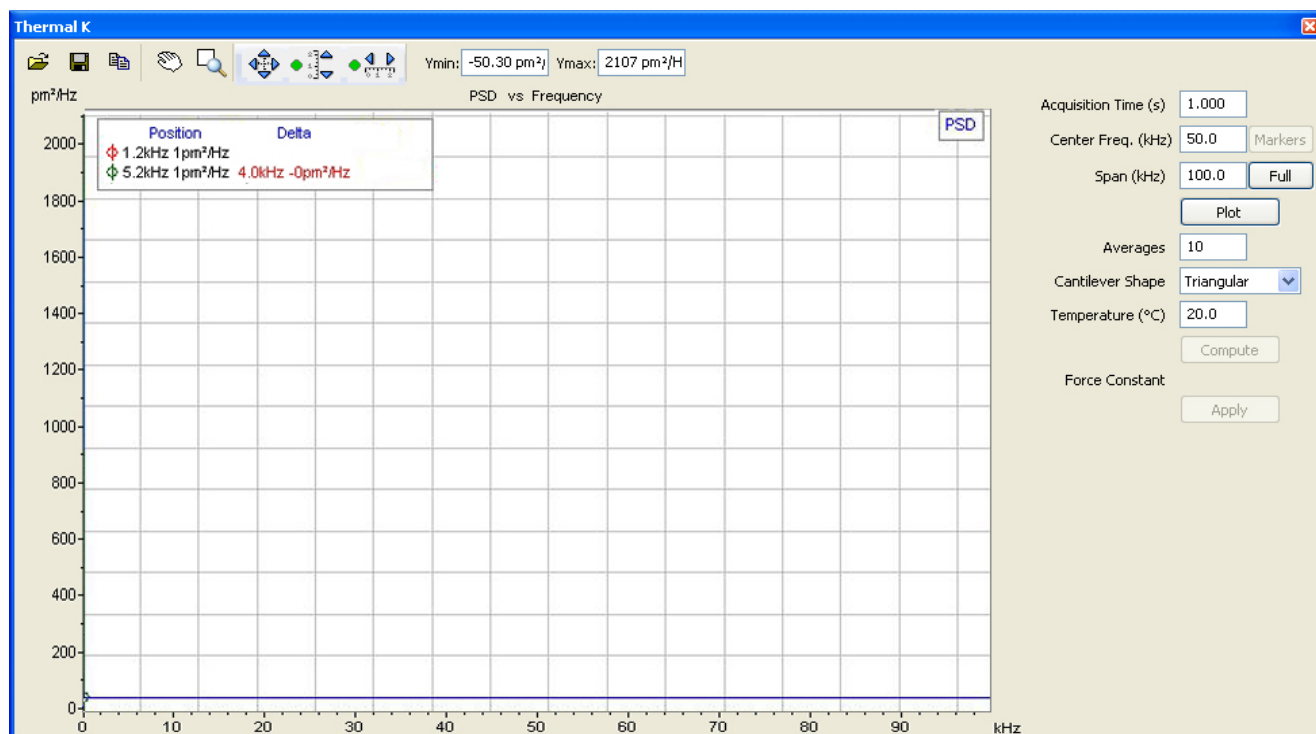


Figure 189 Thermal K plot window

- 11 Set the parameters:
 - a Acquisition Time to **1.0 s**.
 - b Averages to **10**.
 - c Chose the correct cantilever shape from the drop-down menu.
 - d Temperature to the correct ambient temperature in Celsius.
- 12 Click the **Full** button on the right side of the Thermal K window.
- 13 Click the **Plot** button.
- 14 A Thermal K PSD plot (**Figure 190**) should appear.
- 15 Click and drag the red and green circles to define the frequency range in which the resonance frequency should be found. The mouse scroll wheel can be used to zoom in the plot if needed.

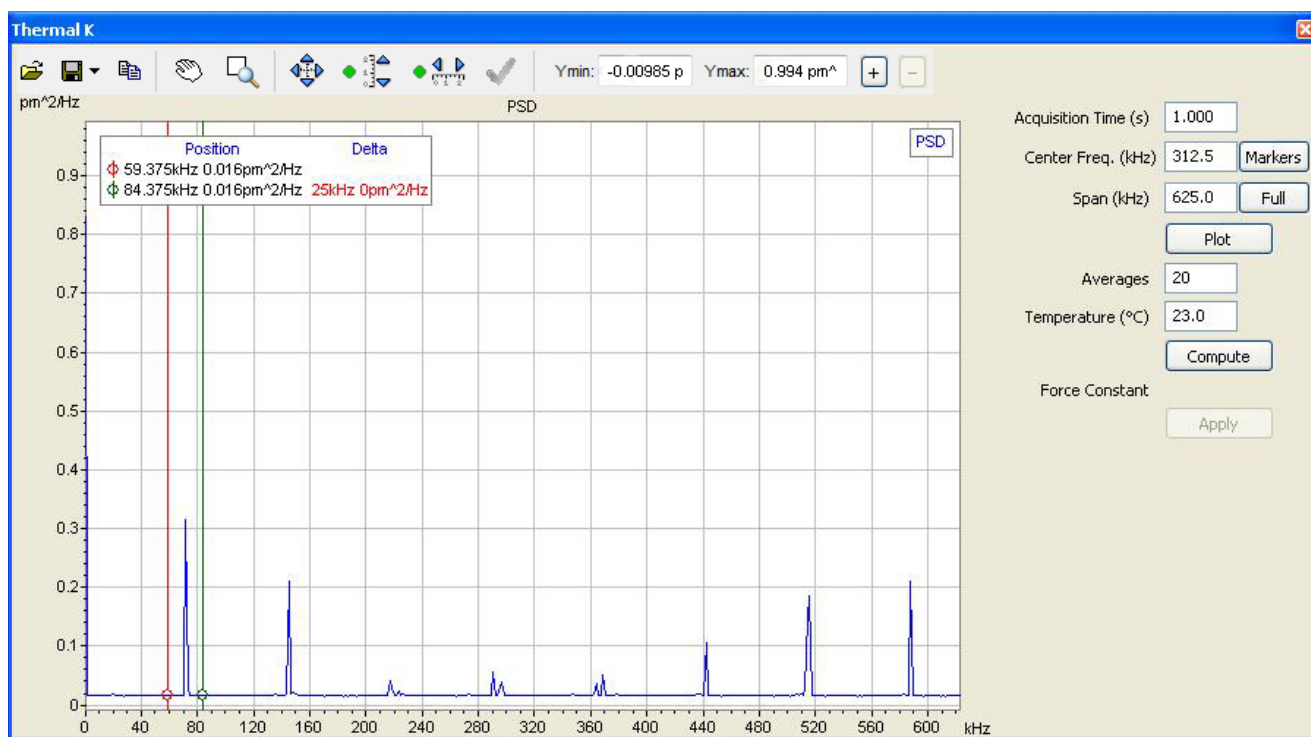


Figure 190 Thermal K PSD plot with markers set to define frequency range

- 16 Click the **Compute** button.

PicoView will acquire the selected number of scans and will average the frequencies within the selected range to complete the calculation. The Thermal K window will refresh, showing the PSD across the frequency range, and a best fit curve in red. The area under the curve is integrated to determine the Thermal K value, which will be displayed as the Force Constant.

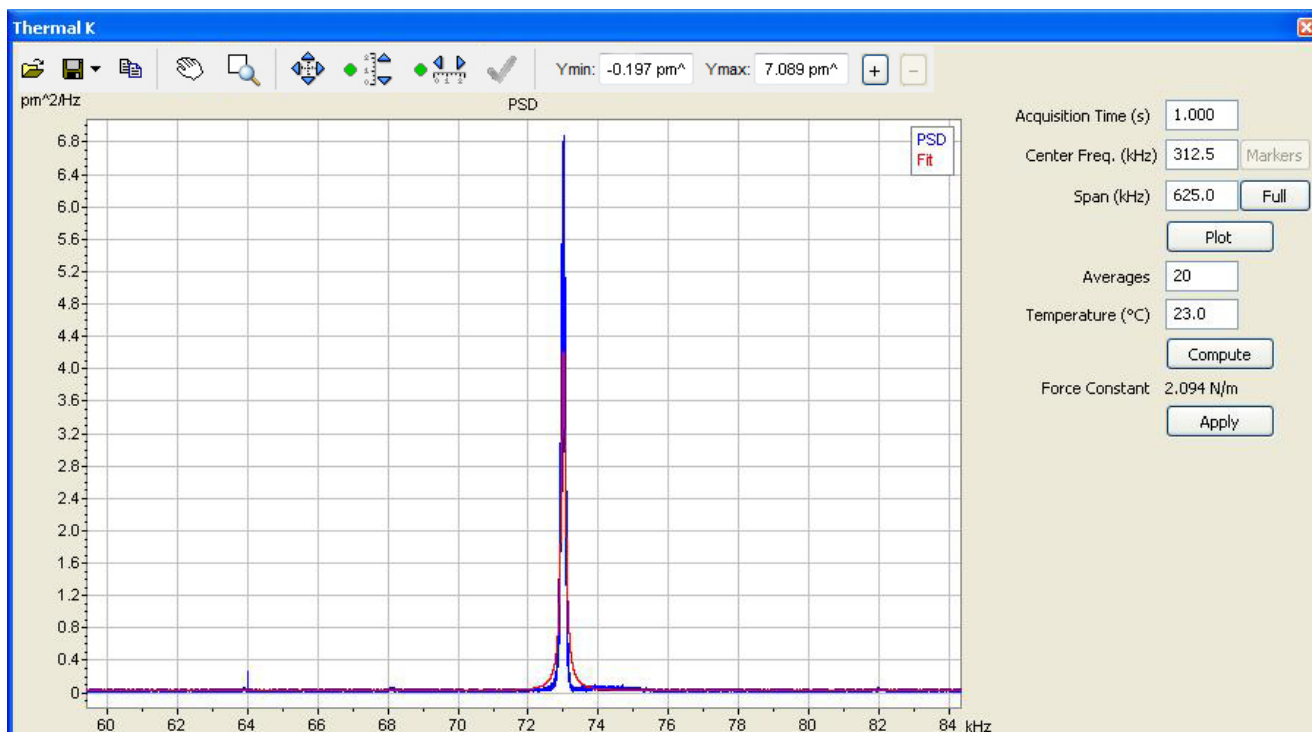


Figure 191 Thermal K window showing best fit curve in red for Thermal K calculation

17 Click the **Compute** button again.

As you accumulate more scans, the Thermal K calculation should converge to a value. Using a small number of scans for this process lets you see how much the calculation is changing as you accumulate data.

18 Once the Force Constant value is stable, click the **Apply** button in the Thermal K window.

The Force Constant should now be updated in the Spectroscopy Advanced tab.

Change the plot window to display Force units if needed:

- 19 In the Spectroscopy plot window, click on the units and select nN from the drop-down menu as shown in Figure 192:



Figure 192 Select units after calibrating the Force Constant

Once the units have been selected, measurements can be taken on subsequent force curves as shown in **Figure 193**:

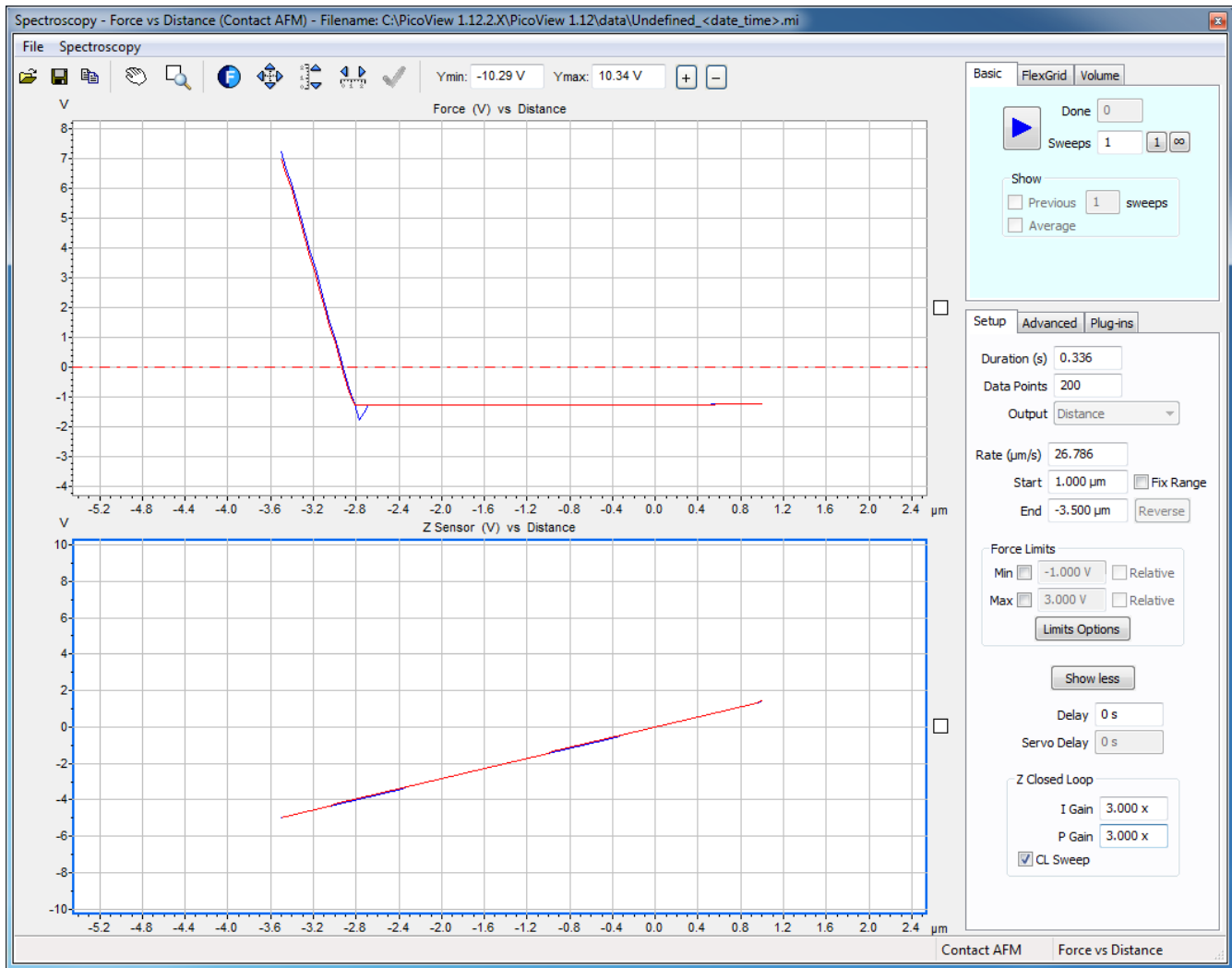


Figure 193 Force curve with calibrated measurements

15 Environmental Control

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Glove Box	292

In addition to the vibration isolation chamber mentioned earlier in this User's Guide, two other options are available to let you control the atmosphere for sample preparation and/or imaging.

Environmental Chamber

The environmental control chamber (Figure 194) lets you isolate samples for imaging in a controlled atmosphere. It can also provide excellent acoustic isolation and protection from air movement, even when atmosphere control is not required. The chamber operates at

atmospheric pressure and is not intended to provide a vacuum or high pressure environment.



Figure 194 Environmental chamber

The environmental chamber includes eight ports which may be used to introduce or remove gases from the chamber, or to allow wiring access for sensors or other electronics. Several types of screw-in fittings are available from Keysight for wires, liquids or 3 mm (1/8 in) inner diameter gas tubing. The ports can be used in any combination. For example, one gas port may be used to introduce a gas while another simultaneously vents the chamber (i.e., when the gas cannot be safely vented directly into the lab). One such example would be a non-aqueous electrochemistry experiment requiring the saturation of an inert as with an organic solvent.

To use the chamber:

- 1 Prepare the sample on a sample plate.
- 2 Mount the sample plate on the microscope.
- 3 Loosen the retaining screws on the front right and left corners of the microscope base and swing the top section up on its hinge.
- 4 Lower the microscope base over the environmental chamber, being careful to avoid contact with the sample plate. The legs of the

microscope base will fit into grooves in the environmental chamber base.

- 5 Swing the microscope back down over the environmental chamber. A groove in the underside of the microscope plate provides a tight seal with the gasket on the top of the chamber (Figure 195).

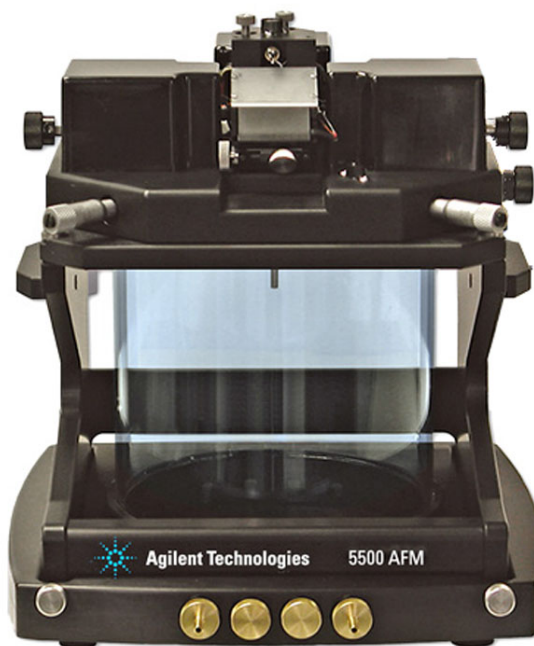


Figure 195 Environmental chamber on microscope

- 6 Secure the environmental chamber to the base plate with the four thumb screws (two in front, two in the rear).
- 7 Tighten the two retaining screws to hold the top plate down.

The chamber also provides an excellent way to displace oxygen from solutions used in electrochemistry experiments. Good results have been obtained by first bubbling an inert gas (nitrogen or argon) through the solution to be placed into the liquid cell, and then setting the environmental chamber up with a steady flow-through rate of 1 to 2 SCFH.

When the microscope and environmental chamber are placed inside the vibration isolation chamber, tubes and cables can be routed to the

environmental chamber through a hole in the side of the vibration chamber.

Glove Box

The glove box lets you create a controlled environment for both sample preparation and imaging. As with the environmental chamber, the glove box includes eight ports for introducing gases, liquids or wires into the chamber. The clear acrylic box is 244 mm (9.6 in) high, 325 mm (12.8 in) wide and 351 mm (13.8 in) deep and can be used at temperatures below 0 °C. The gloves are heavy duty, 15 mil Latex.



Figure 196 Glove Box

The 5500 SPM mounts directly to the top of the box's stainless steel mounting plate, with the stage motor screws and sample plate extending into the box.

16 Electrochemistry

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The electrical potential that exists across the interface between a metal surface and an electrolytic solution is known as the “surface potential.” This is the driving force behind such processes as adsorption, desorption and electron-transfer reactions. Quantifying and controlling this potential is the science of electrochemistry.

Metal electrodes placed into an electrolytic solution will register a net potential composed of two unknown potential drops, one across each electrode-electrolyte interface. A third, chemically reactive reference electrode is maintained in equilibrium with the ions in solution that are oxidized and reduced at its surface. To maintain this equilibrium, the concentrations of reactants must be held constant at the electrode surface, as is true when negligible current flows through the reference electrode.

Figure 197 shows a typical electrochemistry setup, while Figure 198 shows that same setup created with the 5500 SPM liquid cell. Note that

electrochemistry experiments can be conducted using either AFM Modes or STM Mode.

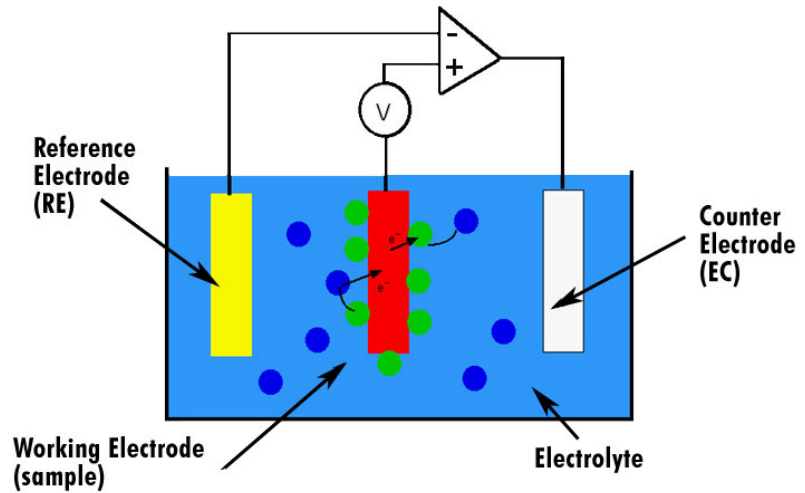


Figure 197 Electrochemistry experiment schematic

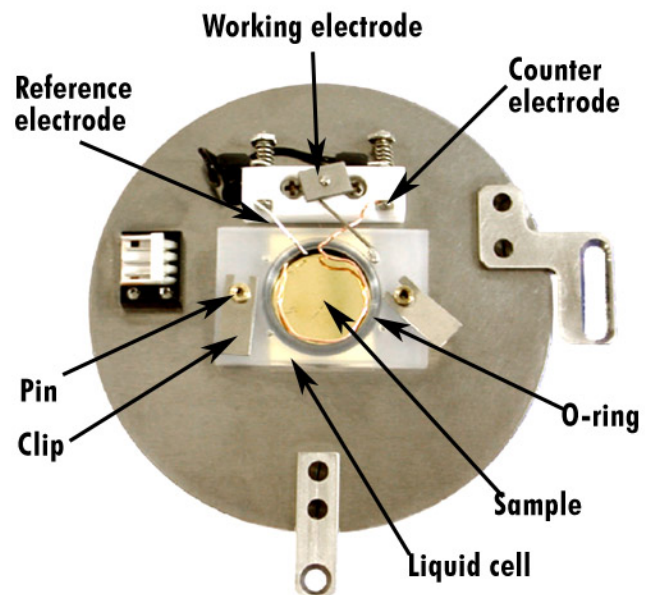


Figure 198 Electrochemistry experimental setup using liquid cell

Equipment

The equipment needed to perform electrochemistry experiments can be as simple as a liquid cell and electrodes, or as complex as a flow-through pump system with a temperature-controlled sample stage. The basic components are described below.

Liquid Cell

The liquid cell, described earlier in this manual, enables imaging in a liquid (Figure 197). The cell is 15 mm (0.59 in) in diameter and seals over the sample with an o-ring. The sample surface must be very flat and larger than the diameter of the cell to avoid leakage.

Electrodes

Three electrodes are typically required for electrochemistry experiments. The experiment being performed will determine the type of wire to be used for the reference and counter electrodes. The type of wire will affect the voltage readings. Prepared electrodes may be purchased from Keysight Technologies, or wires may be formed into appropriate electrodes using the following approximate dimensions.

Working Electrode and Pogo Electrode

Contact between the working electrode (WE) on the sample plate and the sample itself is typically accomplished using the L-shaped pogo electrode included with the system (Figure 199). The pogo contacts the sample through a separate access hole outside of the liquid cell chamber. Since it is not in contact with the electrolyte it does not require special cleaning. A wire made to the same dimensions can be used in place of the pogo.

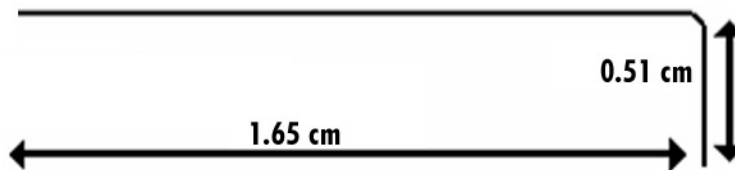


Figure 199 Pogo electrode

Reference Electrode

The reference electrode (RE) should have a diameter of 0.51 mm (0.02 in). It will sit within the electrolyte but will not contact the working electrode (sample).

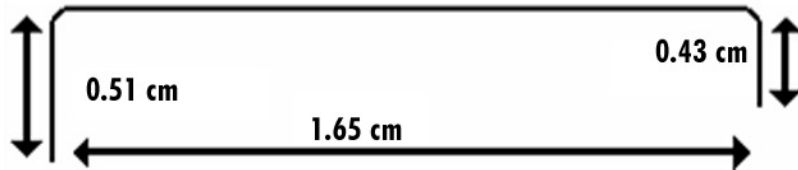


Figure 200 Reference electrode

Counter Electrode

The counter electrode (CE) is typically made from platinum-iridium wire (Figure 201). It should encompass as much of the inner rim of the liquid cell as possible. It may be useful to make the diameter of the electrode slightly larger than the diameter of the cell, so that the electrode will hold itself in place against the walls of the cell.

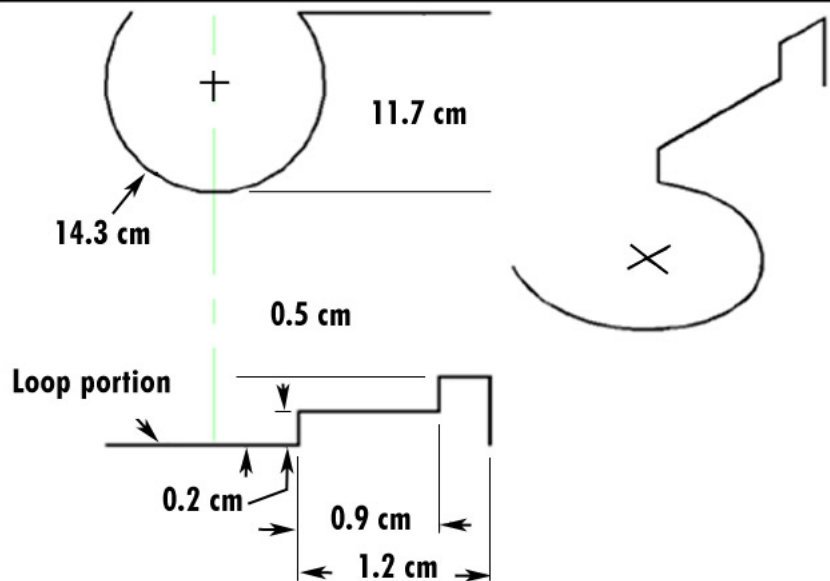


Figure 201 Counter electrode

Cleaning

Thorough cleaning of all components will greatly improve the results of electrochemistry experimentation. Suggestions for cleaning each component are given below.

Liquid Cell Cleaning

The liquid cell should be cleaned prior to every use according to these instructions:

Non-Critical Applications

- 1 Sonicate the liquid cell in laboratory detergent.
- 2 Rinse in 18 M Ω /cm water.
- 3 Rinse in methanol.
- 4 Blow dry under argon or nitrogen gas.

Critical Applications

- 1 Soak overnight in a solution of 70 % concentrated sulfuric acid and 30 % hydrogen peroxide (of 30 % v/v concentration).

WARNING

Use extreme caution when handling this solution. It is a strong oxidizing agent and extremely corrosive.

- 2 Rinse thoroughly, at least four times, in 18 M Ω /cm water.
- 3 Boil for one hour in 18 M Ω /cm water, changing the water every 15 minutes. You may instead rinse overnight in 18 M Ω /cm water.
- 4 Rinse two more times in 18 M Ω /cm water.
- 5 Dry under argon or nitrogen gas.

Electrode Cleaning

Electrodes should be carefully cleaned prior to assembling the liquid cell. This may even include flame annealing of the electrodes prior to use in certain cases.

Sample Plate Cleaning

Since the sample plate does not directly contact the sample surface or electrolyte, a general cleaning with methanol or ethanol prior to assembly is sufficient.

Substrate Cleaning

Substrates should be free of surface contaminants. Gold substrates should be hydrogen flame annealed prior to imaging for best results.

Assembling and Loading the Liquid Cell

It is recommended that the assembly procedure be carried out in a laminar flow hood, glove box or other clean environment. The work surface should be well cleaned prior to assembling, and gloves should be worn to prevent contaminating the electrodes and liquid cell. Refer to [Figure 198](#) for a view of the components described below.

- 1 Place a clean substrate onto the sample plate.
- 2 Push the liquid cell onto the spring-loaded pins on the sample plate. Verify that the O-ring is in contact with the substrate at all points.
- 3 Push the spring-loaded pins up from the bottom to expose the pin slots.
- 4 Insert the cell clamps into the slots and release the pins to hold the liquid cell in place.
- 5 Place the pogo electrode into the hole in the wall of the liquid cell nearest to the working electrode clamp.
- 6 Push the working electrode clamp up from the bottom and place the end of the pogo under the clamp. Let the clamp spring back to hold the electrode.
- 7 Use a multimeter to check for good conductivity between the sample and the working electrode clamp.

NOTE

For improved conductivity an additional wire can be used to connect the working electrode to the substrate through another hole on the liquid cell.

- 8 Insert the counter and reference electrodes into the sample plate block (Figure 198). Push the spring-loaded clamp forward from behind, insert the electrode and release the clamp.
- 9 Position the electrodes so that they will make good contact with the electrolyte but will not touch the sample substrate or each other.
- 10 Use a multimeter to check for good conductivity between the reference electrode and sample plate clamp, and between the counter electrode and sample plate clamp.
- 11 With the multimeter verify that the reference and counter electrode are not shorted to another electrode or to the substrate.
- 12 Verify that the AFM probe or STM tip will pass through the electrodes without any contact.
- 13 Fill the liquid cell enough to submerge the sample. Check for leaks and reposition the cell if necessary.
- 14 Verify that the counter electrode feedback is turned off in the software and that the potentials are set appropriately for the particular experiment.
- 15 Connect the sample plate to the microscope using the 3-pin EC cable connector of the EC/MAC Cable.
- 16 Load the sample plate onto the microscope.
- 17 Approach the sample.

Troubleshooting

The most frequently encountered problem is leakage from around the bottom of the liquid cell. It is generally more of a problem with solvents that “wet” the substrate well, such as methanol. This causes leakage current and erratic imaging. Make sure that the sample is flat and large enough to fit underneath the liquid cell without gaps. Check that the O-ring is clean and pliable. Tightening the sample plate screws will increase pressure on the cell. Do not overtighten as this may crack the liquid cell.

Electrochemistry Definitions

- 1 In STM Mode the tip is always virtual ground. Virtual ground means that the potential of the probe is actively kept at ground by the


operational amplifier, but the tip itself is not connected physically to ground.

- 2 In AFM Mode the potential of the tip is determined by the setting of the switch on the back of the Head Electronics Box (for conductive cantilevers):
 - a If the switch is set to **WE** then the cantilever is biased to the same potential as the working electrode (sample substrate).
 - b If the switch is set to **Tip** then the cantilever is tied to the tip bias DAC output. This is typically ground unless the bias setting is configured so that the probe is biased instead of the sample.
 - c If the switch is set to **BNC** then the cantilever potential is the same as the Cantilever BNC potential. This can be driven externally or will float if nothing is connected to the BNC.
- 3 Sample Bias = WE.
- 4 Sample Potential = WE - RE.
- 5 Probe Potential = - RE.
 - a Sample Potential = Sample Bias + Probe Potential.
 - b The only two independently-controlled potentials are WE and RE.
- 6 $VEC = WE - RE$
- 7 IEC = Current into or out of the working electrode. Positive is flowing into the working electrode.

Software Controls

The procedure below is a general guideline for how to use the Electrochemistry window in PicoView. Additional details regarding the

menu, toolbar and main tab features are in the online PicoView User Guide.

Choose **Controls > Electrochemistry** from the PicoView main window, or **click** on the Electrochemistry icon  from the toolbar button to open the Electrochemistry window (Figure 202).

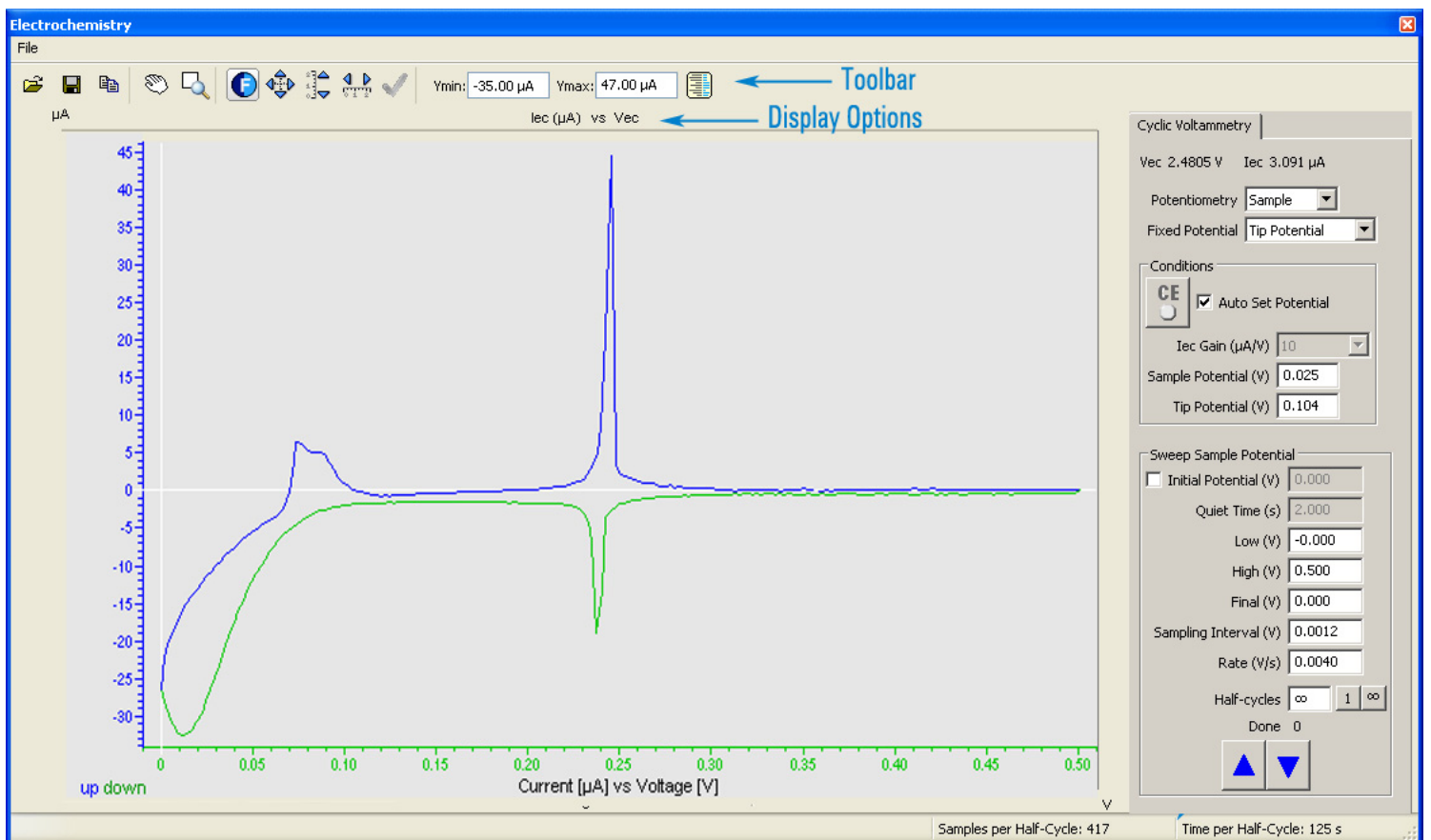


Figure 202 Electrochemistry window overview

The Display Options can be adjusted by clicking on the option and selecting from the drop-down menu as shown in [Figure 203](#).

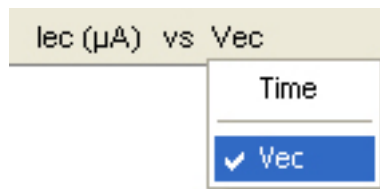


Figure 203 Select Display Options for the Electrochemistry window

Guidelines for Electrochemistry

The following is a general procedure for using the Electrochemistry window in PicoView. The user will need to know what electrodes and solution to use. The choice of when to image the sample is also dependent upon the experimental requirements.

- 1 Choose **Controls > Electrochemistry** to open the Electrochemistry window.
- 2 Select the **Auto Set Potential** check box.
- 3 **Click** the CE button to turn the counter electrode On. The button should be green.
- 4 Set the **Iec Gain** from the drop-down menu. Initial values are typically 1 or 10.

Note for older systems this is controlled from the HEB.

- 5 Set the Sweep Sample Potential parameters. These are sample dependent.
- 6 Select sweeps for 1 cycle or continuous cycling .
- 7 Once all parameters are set, begin the voltage sweep by clicking the blue triangle **Start** button to sweep up or down.

To save successive electrochemistry measurements, have files saved automatically after each set of potential sweeps:

- 1 In the Electrochemistry window, choose **File > Set filename**
- 2 In the **Save As** dialog box, enter a filename for the data set. Note the file type will default to *.mi.
- 3 Click the **Save** button.
- 4 Turn on Auto Save by choosing **File > Auto Save Every Experiment**. The title bar at the top of the window will indicate Auto Save is on, circled in red in [Figure 204](#). It will also be indicated by a check mark next to Auto Save in the Electrochemistry File menu.

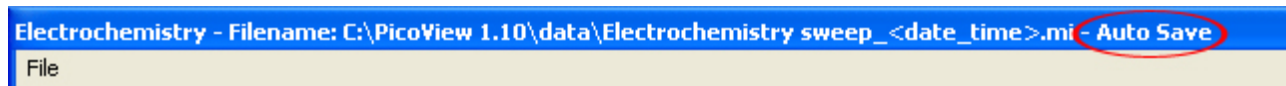


Figure 204 Auto Save turned on for Electrochemistry experiments

Appendix A

Wiring Diagrams

Keysight 5500 SPM Standard Wiring Diagram	305
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Keysight 5500 SPM with MAC Mode, Force Modulation Imaging	307
Keysight 5500 SPM with MAC III Option	308
Keysight 5500 SPM with MAC III Option and Closed Loop Scanner	309

The following pages contain wiring diagrams for several common configurations of the Keysight 5500 SPM.

Keysight 5500 SPM Standard Wiring Diagram

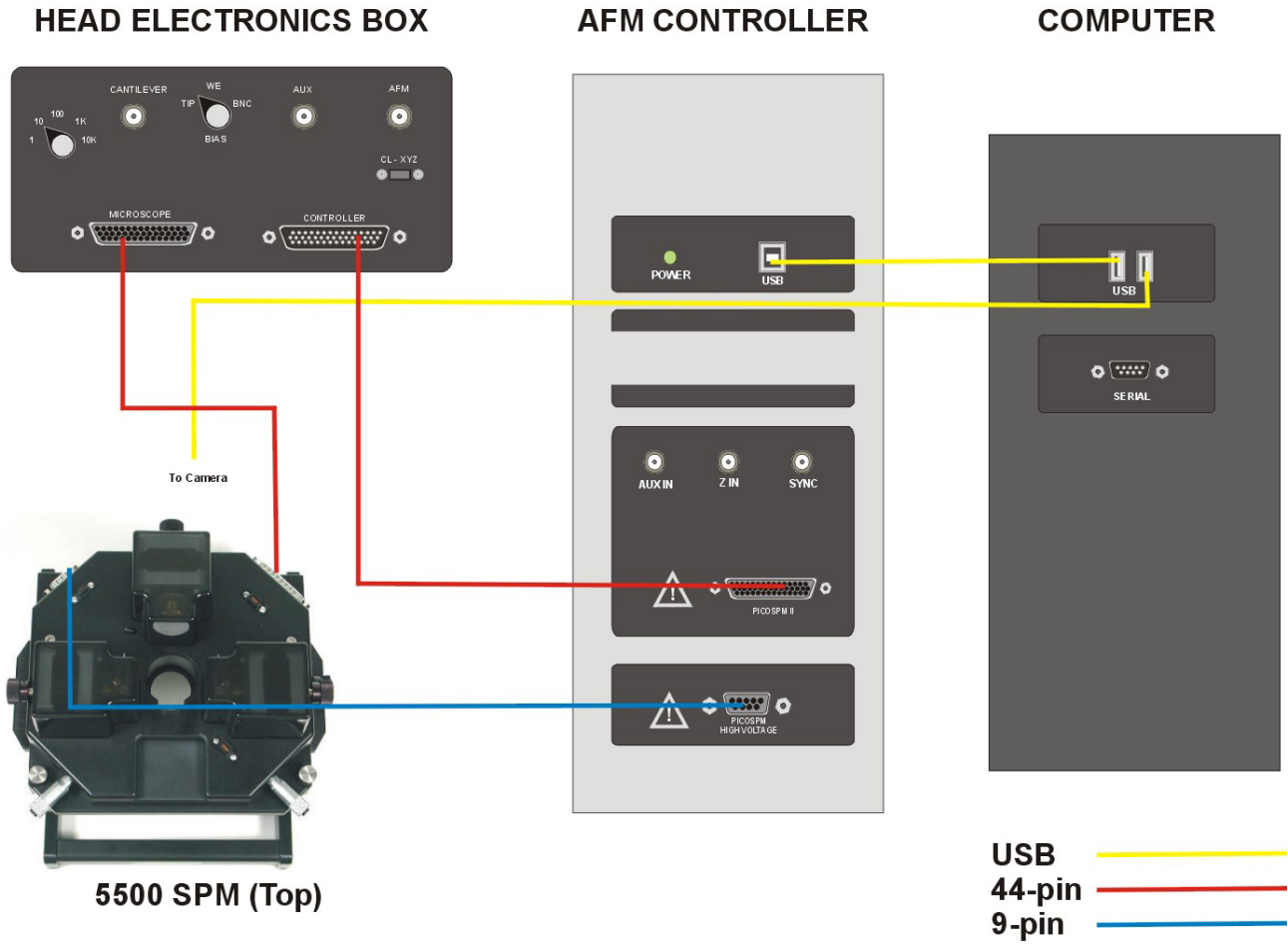


Figure 205 Wiring diagram for Keysight 5500 standard configuration

Keysight 5500 SPM with MAC Mode Controller

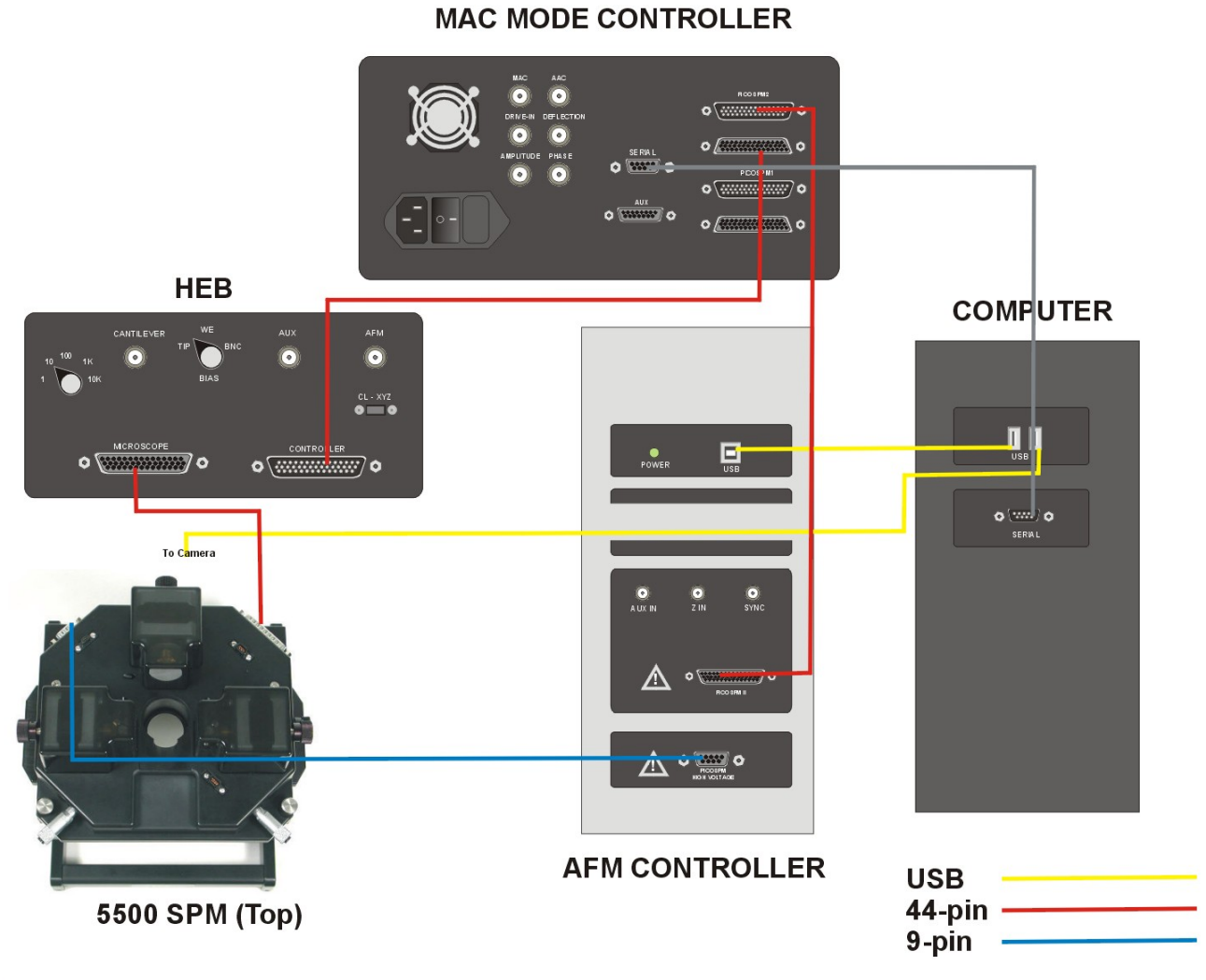


Figure 206 Wiring diagram for Keysight 5500 SPM with MAC Mode Option

Keysight 5500 SPM with MAC Mode, Force Modulation Imaging

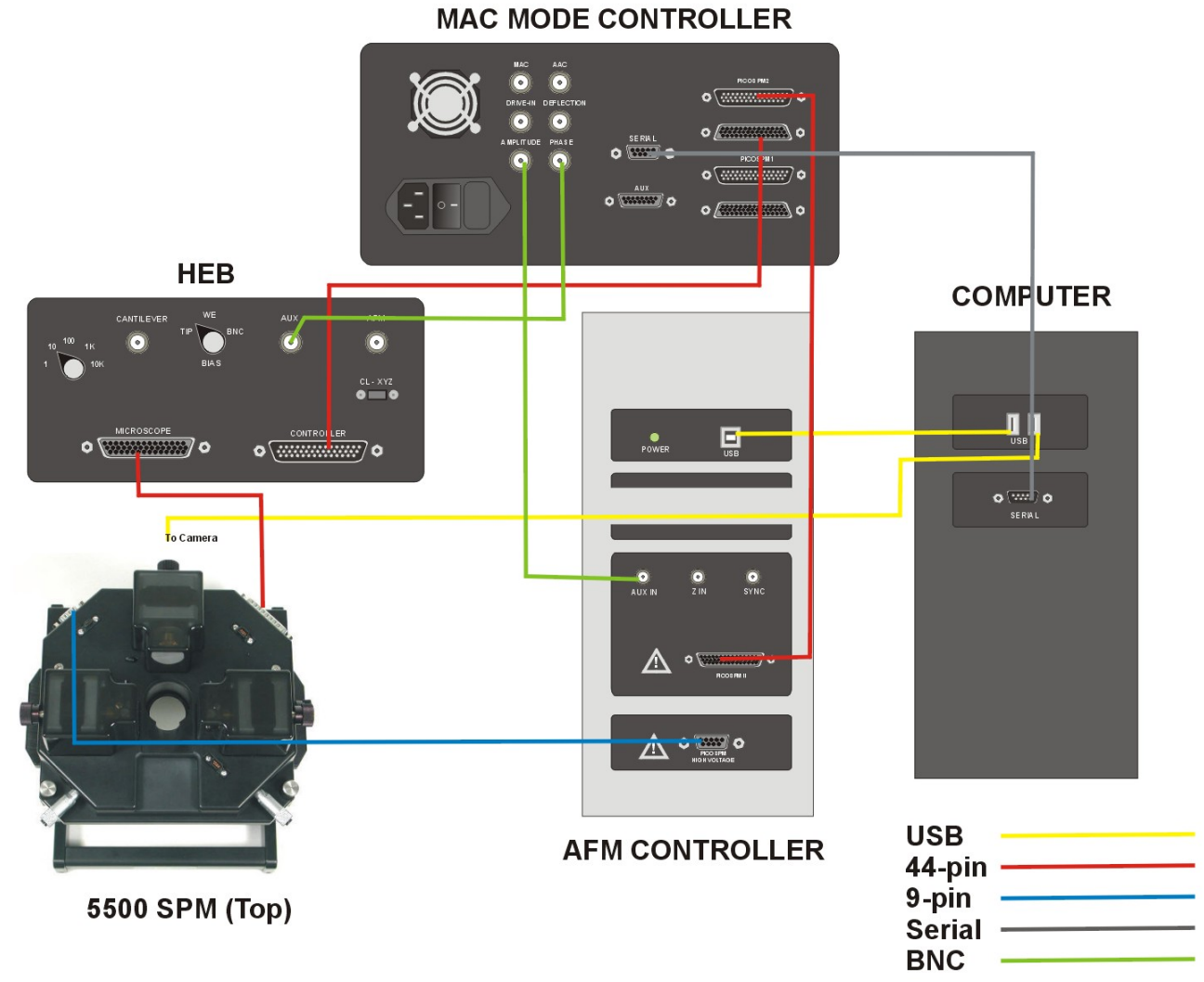


Figure 207 Wiring diagram for Keysight 5500 SPM with MAC Mode Option, Force Modulation imaging mode

Keysight 5500 SPM with MAC III Option

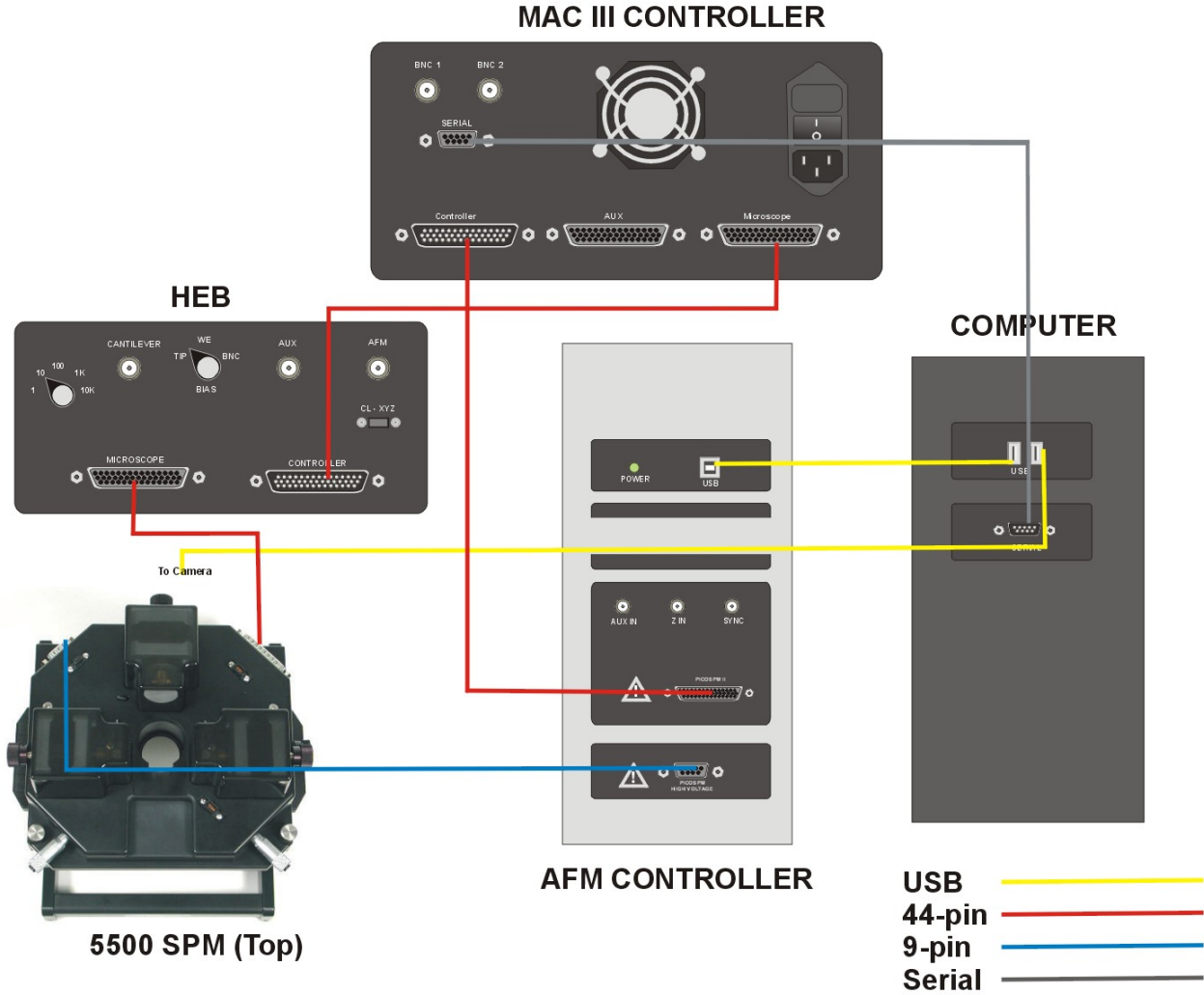


Figure 208 Wiring diagram for Keysight 5500 SPM with MAC III Option

Keysight 5500 SPM with MAC III Option and Closed Loop Scanner

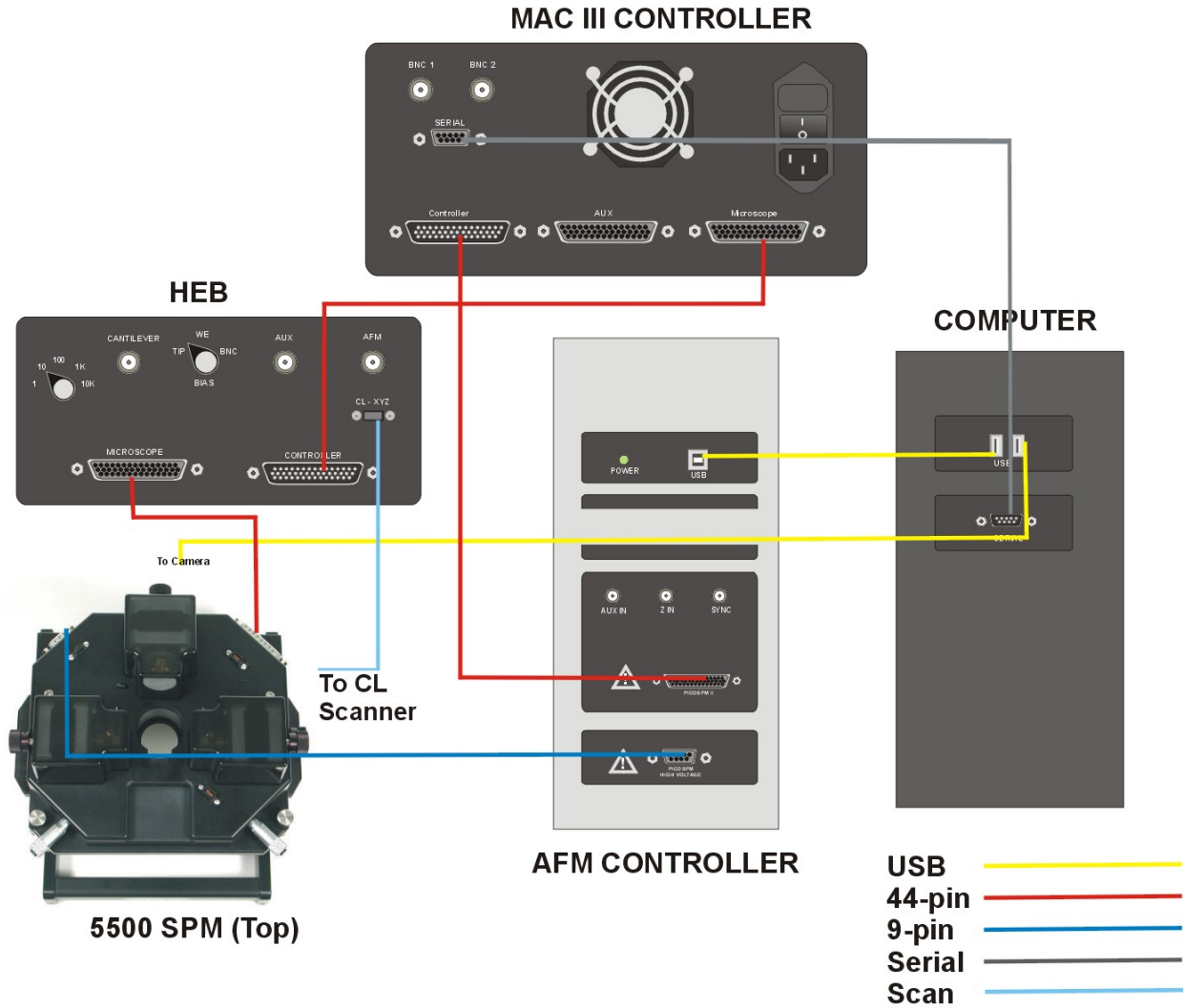


Figure 209 Wiring diagram for Keysight 5500 SPM with MAC III Option and Closed Loop Scanner option

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5500 SPM User's Guide

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