

# AFM WORKSHOP™

## HR - AFM

Users Guide V1.0



# Definitions and Symbols

The following terms and symbols are used in this document and also appear on the product where safety-related issues occur.

## General Warning or Caution



[Image Def1: exclamation symbol]

The exclamation symbol may appear in warning and caution tables in this document. Symbol designates an area where personal injury or damage to the equipment is possible.

## European Union CE Mark



[Image Def2: CE symbol]

The presence of the CE Mark on AFMWorkshop equipment means that it has been designed, tested and certified as complying with all applicable European Union (CE) regulations and recommendations.

## Warnings/Cautions/Notes

The following are definitions of the warnings, cautions and notes that may be used in this manual to call attention to important information regarding personal safety, safety and preservation of the equipment, or important tips.

### WARNING

Situation has the potential to cause bodily harm or death.

### CAUTION

Situation has the potential to cause damage to property or equipment.

### NOTE

Additional information the user or operator should consider.

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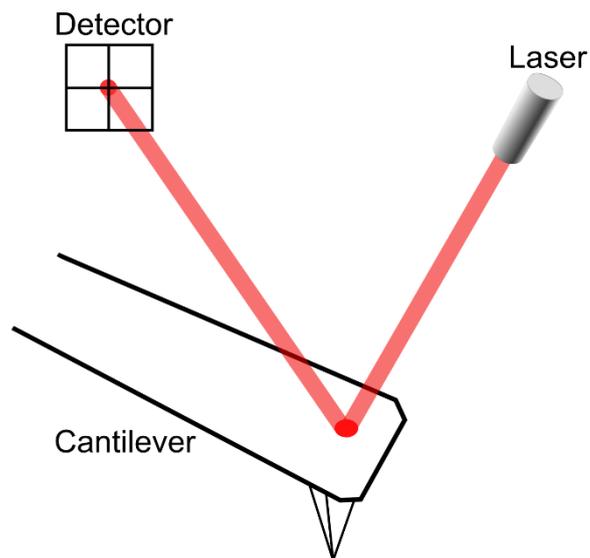
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## 1. Introduction to Atomic Force Microscopy

In an AFM (atomic force microscope), a probe is scanned over a surface and the motion of the probe is monitored to create a three-dimensional image of the surface. These unique instruments are capable of measuring high-resolution images in both ambient air and liquids, accurately determine the dimensions of surface features of only a few nanometers in size.

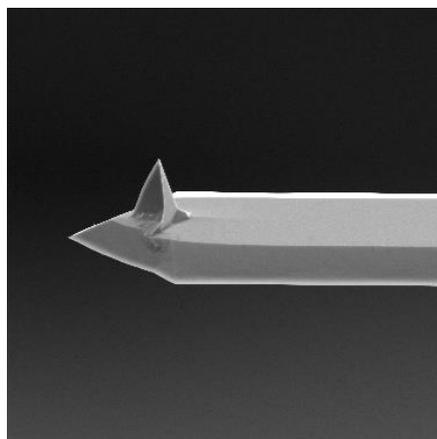
The three-dimensional motion of the sample (or probe) is generated by piezoelectric ceramics. These sensitive ceramics allow motions as small as a fraction of a nanometer. Typically, the sample (or probe) is moved in a raster pattern as the probe moves across the surface.



[Image1.1: Light Lever]

A light lever sensor is used for controlling the force of the probe on the surface while the sample is scanned. The light lever reflects a laser beam off the surface of a cantilever into a photo-detector. As the probe interacts with a surface, the cantilever deflects, and this motion is sensed by the photo-detector.

With this light lever, measurement and control of forces as small as a pico-newton are possible. With such small forces, very small probes may be used. With micro-machining methods, probes can have diameters of only a few nanometers.

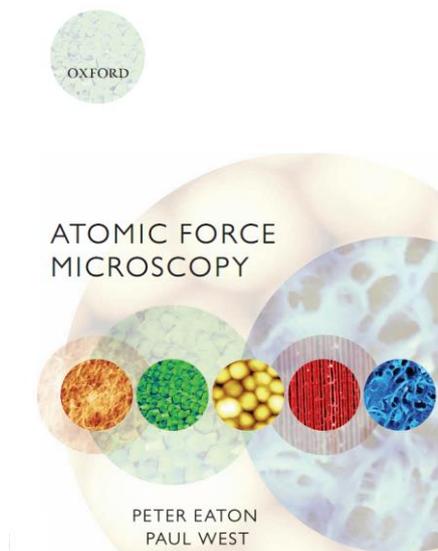


[Image1.2: SEM image of AFM Probe]

The light lever can be made more sensitive by vibrating the cantilever with a small piezoelectric ceramic. When the vibrating probe interacts with the surface, the amplitude of vibration may be monitored and used to control the probe's force on the surface.

Modern atomic force microscopes include not only a probe and piezoelectric scanner, but additional hardware for bringing the probe rapidly into the proximity of a surface. A video optical microscope is very helpful for operating an AFM. The video microscope helps with aligning the light lever and probe approach, and for finding features for scanning.

For an in-depth description of AFM instrumentation, we recommend the book Atomic Force Microscopy by Peter Eaton and Paul West. This book provides a complete theoretical and practical explanation for the design and application of AFMs.



[Image1.3: Book “Atomic Force Microscopy” by Peter Eaton and Paul West]

## 2. Introduction to the HR-AFM

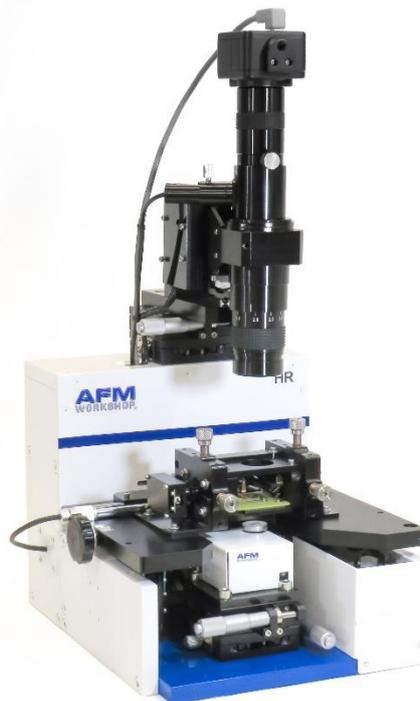
When fully assembled, the HR-AFM comprises four subunits. They are the control computer, the Ebox, the stage, and the video optical microscope.

### 2.1 Stage

Samples are held and scanned on the AFM stage. The stage also includes a translator system which moves the light lever force sensor in a vertical direction. The stage also includes the light lever, precision XY translators, and the piezoelectric scanner. Samples are held magnetically on a 1" diameter plate at the top of the piezoelectric scanner.

Optimal images are measured with the AFM stage if it is in a vibration and acoustic noise-free environment. If necessary, a vibration and acoustic isolation system should be used. AFMWorkshop may have also supplied these components with the system, or the user can obtain their own. Appendix A provides more information on the best location for the AFM stage.

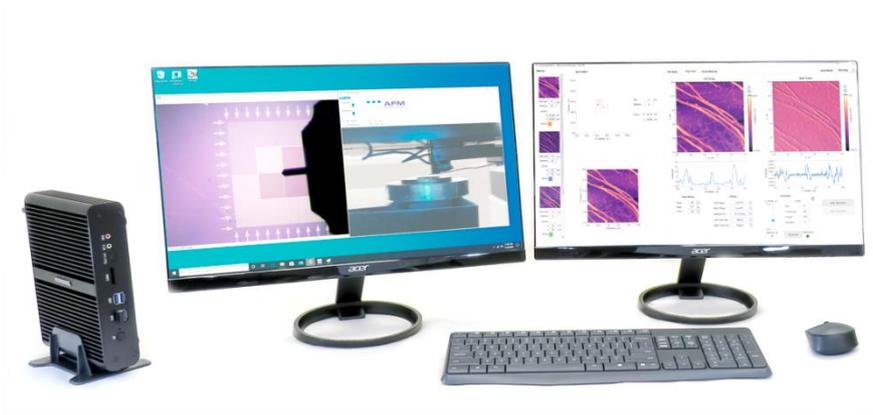
On the back cover of the stage is a modes connector. Signals required for implementing additional modes such as conductive AFM, STM and EFM are provided. Additional information on the cable configuration is provided in Appendix A.



[Image 2.1: HR-AFM stage combined with the video microscope]

### 2.2 Computer

The control computer is a standard PC-type computer with a Microsoft Windows operating system. There are two programs required to operate the HR-AFM: the AFM control software and the software for the color video camera.



[image2.2: control computer]

### 2.3 Ebox

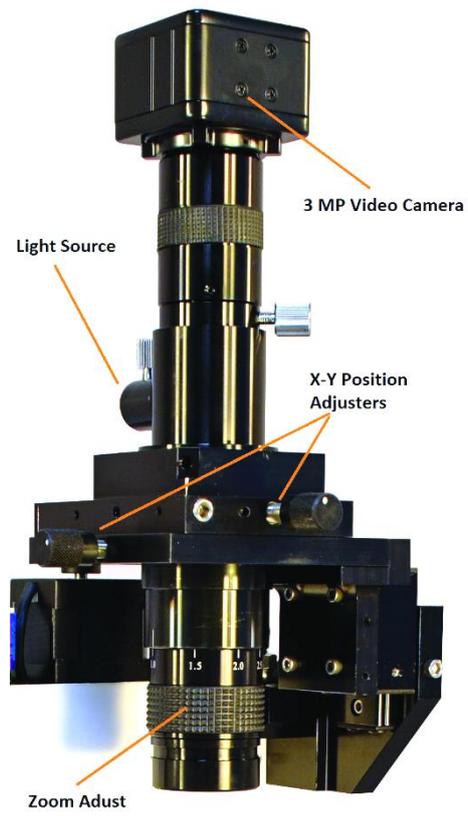
The Ebox sends and receives signals from the computer through a single USB cable. Electronic signals are then sent to the stage through a 60-pin ribbon cable. Additionally, a grounding cable is connected between the stage and the Ebox. All cables are connected at the rear of the Ebox. In addition to the cable to the stage, there is a plug for an auxiliary 50-pin ribbon cable that gives access to the Box's internal electronic signals.



[Image2.3: Front and back views of the HR-AFM Ebox showing indicator lights and connectors for ribbon cables and USB]

### 2.4 Video Optical Microscope

Aligning the light lever force sensor is greatly facilitated by the aid of the video optical microscope. The video microscope also helps to locate features on a surface for scanning. With the aid of the video microscope, tip approach can be executed much more rapidly. Images from the video microscope are displayed on the computer's video monitor.



[Image 2.4: Video microscope with XY translators and focus adjust]

### 3. Software

The HR-AFM includes three separate software modules in the AFM Installation files: AFMWorkshop AFM Control Software, Workshop Video Microscope Software, and Gwyddion Image Analysis Software. All the software packages require a system running a Microsoft Windows OS.

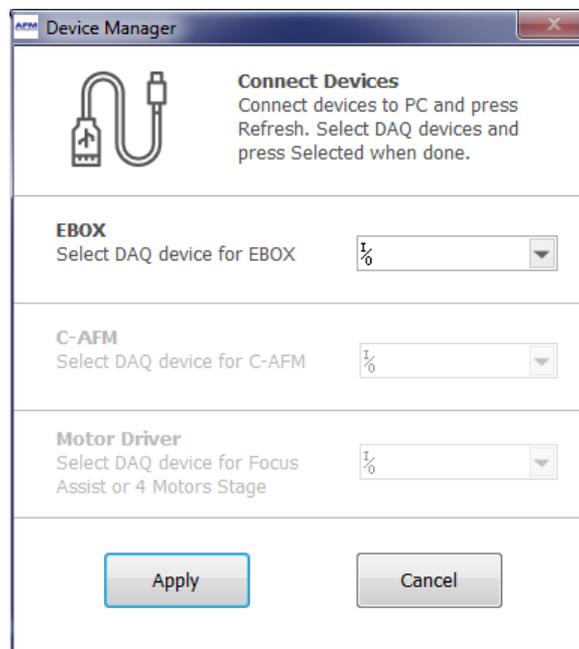
#### 3.1 Installation Procedure

**NOTE:** If the target computer has a previous version of LabVIEW installed, it is imperative that it first be completely removed.

Locate the provided AFM installation files for the USB camera, Gwyddion, and AFMWorkshop control software and follow the prompts. The user may be asked to input his/her scanner size, so that the setup procedure will copy the files that correlate with that scanner size onto the desktop. Reboot when directed. All instructions can be found in the readme file.

**NOTE:** A Windows Security pop-up may appear when installing the Video Microscope driver that states that Windows cannot verify the publisher of the driver software. Choose the option to “Install this driver software anyway.”

Upon initializing the AFM Control Software for the first time, the user will be asked to confirm the serial number of the National Instruments DAQ card in the Ebox. Typically, there will only be one card option available. The software will display this pop-up every time the user plugs in a different Ebox, or reinstalls the software.



[Image 3.1: DAQ Module Selection]

### 3.1.1 Gwyddion Image Analysis Software

Gwyddion is open-source software and the latest version of this image-analysis software is freely available on the Internet at: <http://gwyddion.net/>. The functions of the Gwyddion image analysis software are:

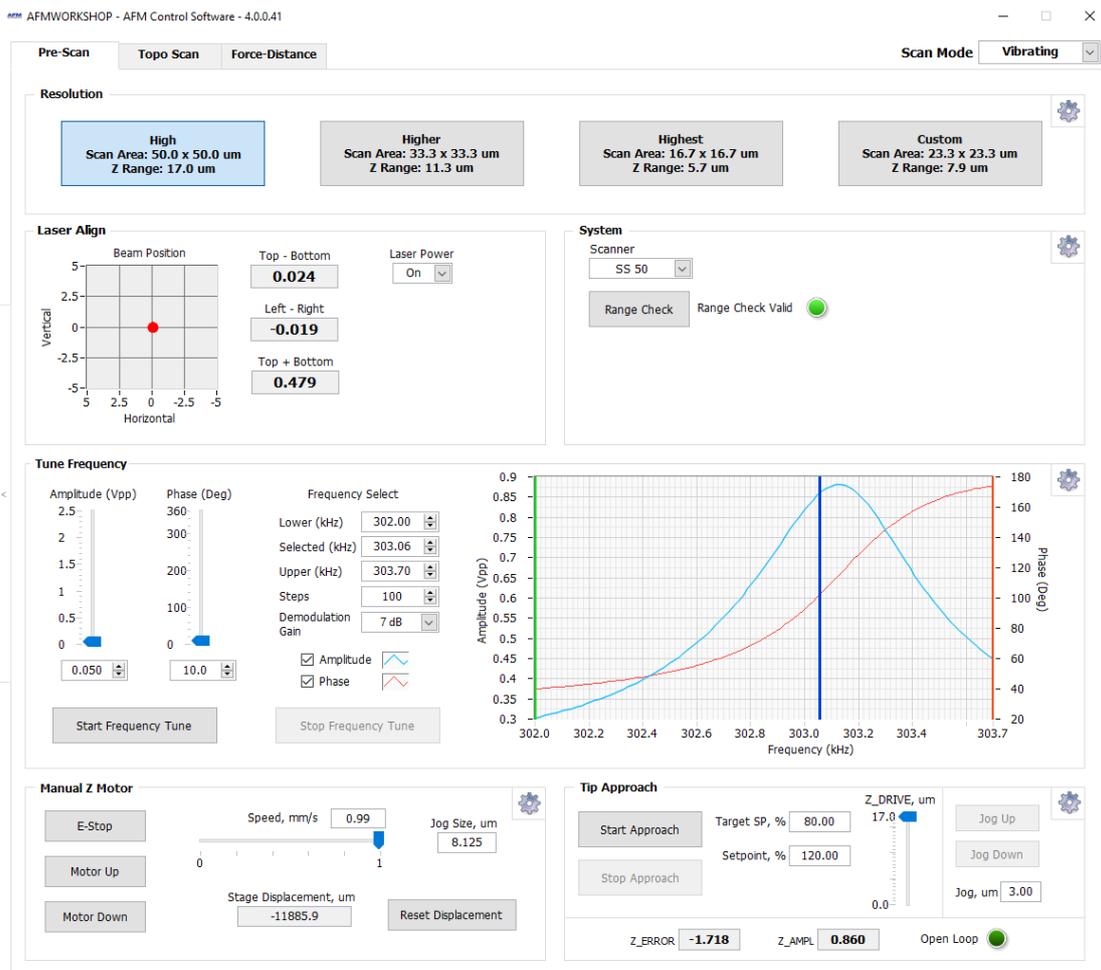
- Processing routines such as leveling, deglitching, filtering, and smoothing which alter the images.
- Display functions which change how the data is viewed, including 2-D, 3-D, light shading, and color mapping.
- Analysis options that are used for obtaining quantitative measurements from images, such as line profiling, particle analysis, and histogram analysis.

### 3.2 AFM Control Software

Once launched, the AFM Control Software has a collapsible navigation area to the left, and three (or more) tab-screens that can be viewed by pressing the tabs at the top edge of the screen. The first tab is for the Pre-Scan window (section 3.2.1) and the second tab is for the Topo Scan window (section 3.2.2). These two tabs along with sample navigation area (section 3.2.3), are all that are needed for measuring AFM images. There is a third tab that, when activated, permits force-distance curve measurements (section 3.2.4). If additional modes are installed on the system (e.g. magnetic force microscopy or lithography), there may be more tabs available.

#### 3.2.1 Pre-Scan Window

The Pre-Scan window contains all of the functions that require adjustment before an image is measured. Normally, it is used before any other parts of the software, to set up the instrument as required, and approach the probe to the sample surface. In this window, the various controlled are organized in the order in which they are normally used, top-to bottom and left-to-right.



[Image 3.2: Pre-scan Window]

### 3.2.1.1 Modes

There are two image scanning modes: Vibrating mode and Non-Vibrating (contact) mode. The modes are selected with the Scan Mode button. When Vibrating mode is selected, the frequency sweep window is activated. When Non-Vibrating mode is selected, the frequency sweep window is deactivated.

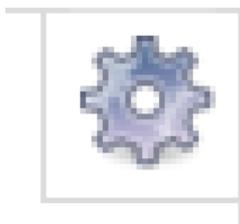
### 3.2.1.2 Resolution Modes

The HR-AFM is designed with care in order to enable the highest-possible imaging resolution. In order to make this process simple, The first set of controls in the Pre-scan tab allow the user to choose from instrument settings enabling “High”, “Higher”, to “Highest” resolution and a “Custom” mode. These controls allow the user to choose a trade-off between large scanning area areas and ultimate resolution. The “high” resolution mode will limit the ultimate resolution of the instrument, but allows for large area scanning (depending on the scanner used). While the “highest” resolution mode has great resolution, but limits the area and height of features that can be scanned. The “custom” mode allows the user to set up their own gain settings, for maximum flexibility. To understand better what these settings do, see section 6.

**NOTE:** The “High” mode really does allow for high-resolution imaging, and can be successfully applied to image features as small as 5 nm. In addition, this mode is the simplest to use, and allows for large AFM scans. Therefore, unless the user is

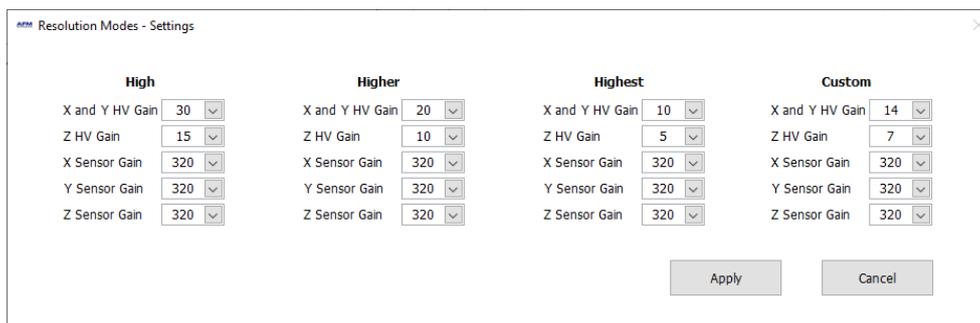
expecting to measure features below 5 nm in sizes, it is recommended to use the “High” resolution mode.

**NOTE:** As a convention, each control cluster in the AFM Control Software has a **settings** window for advanced controls, or to change the way the other controls work, where available. This can be accessed by pressing the gear in the top right hand corner of the panel.



[Image 3.3: Gear icon to access advanced controls]

**Settings:** The settings window for the Resolution modes, can be accessed by clicking the gear icon. This window allows changing various setting for each of the four available modes. In general, the user will want to change only the X and Y HV Gain, and the Z HV Gain values. We recommend that only the “Custom” values are changed, and the “High”, “Higher” and “Highest” values are left at their default values, which are shown below for reference.

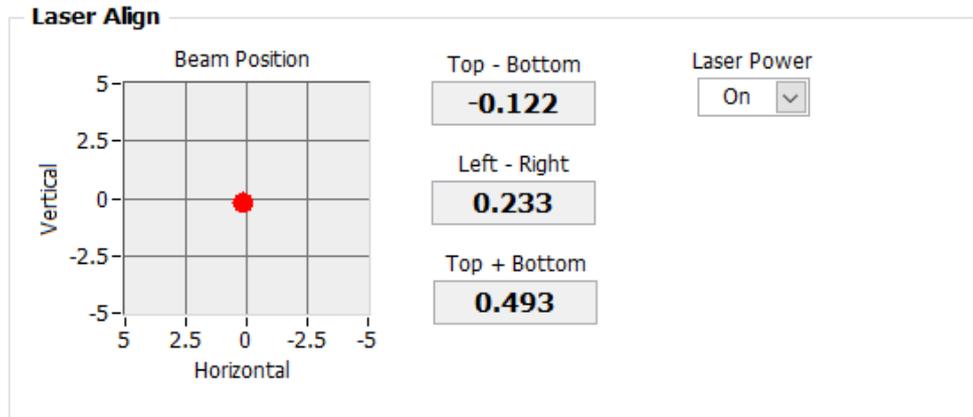


[Image 3.4: Resolution modes setting window]

### 3.2.1.3 Laser Align

The position of the laser on the four-quadrant photo-detector is presented numerically and visually in the Laser Align window. These two indicators are both updated in real time and are used for aligning the light lever. There is a switch for turning the laser On and Off. In addition, the “Top + Bottom” signal is displayed which is the total laser signal registered by the photodetector.

**NOTE:** After the HR-AFM light lever is set up for the first time (see Appendix A.6), the thumb screws used for laser alignment should not need to be turned more than a few turns.



[Image 3.5: Laser Align panel]

### 3.2.1.4 System Panel

The system panel has a drop-box for selecting the scanner to be used by the HR-AFM. The HR-AFM instrument uses a sample-scanning configuration. The types of scanner that can be selected are:

- SS 15: This is a high resolution scanner that has a range of  $15 \times 15 \mu\text{m}$  in X and Y and  $7 \mu\text{m}$  in Z. Unlike the other sample scanning scanners, the SS 15 scanner has no position sensor in the Z axis.
- SS 50: This is a highly flexible mid-range scanner that has a range of  $50 \times 50 \mu\text{m}$  in X and Y and  $17 \mu\text{m}$  in Z. The SS 50 scanner has position sensors in all three Z axes.
- SS 100: This is a large-range scanner that has a range of  $100 \times 100 \mu\text{m}$  in X and Y and  $17 \mu\text{m}$  in Z. The SS 100 scanner has position sensors in all three Z axes.

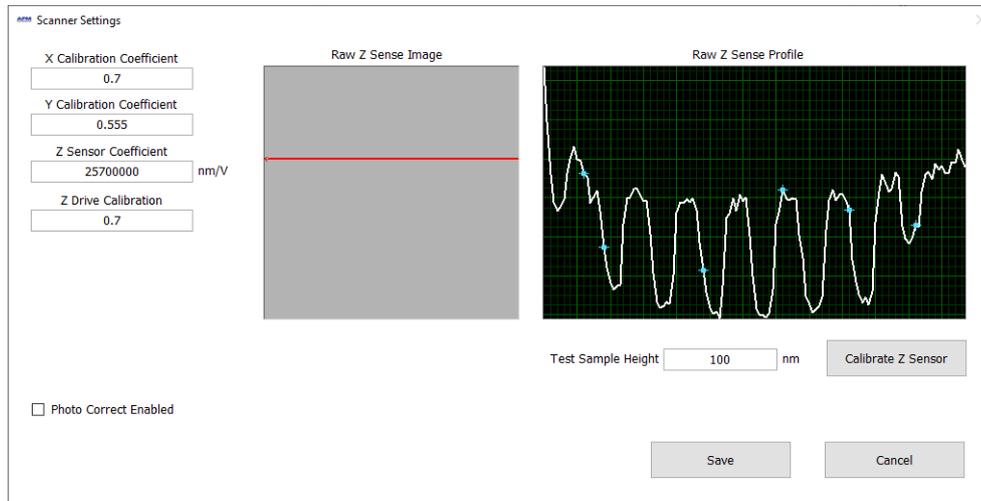
Just below the drop-down box for selecting the scanner is the Range Check button. The usable range of the piezoelectric scanner is measured with the Range Check option. When started, the scanner is moved in a square pattern (in X and Y), which can be readily observed with the video optical microscope, and then up and down in Z. This function must be completed before beginning a scan. A range check **must** be performed every time a new scanner is selected, or the Resolution mode is changed. Until the range check is performed, and the “Range Check Valid” light is shown, the AFM Control Software will not allow scanning to take place.



**CAUTION:** Before activating the Range Check function, make sure that the probe is moved away from the sample surface. If the probe is too close to the surface, the probe may be damaged.

**Settings:** The setting window for the System frame is shown below. This window allows the user to calibrate the selected scanner. Appendix C explains how to calibrate the HR-AFM in detail.

**NOTE:** Changing the calibration values will change the scale on images measured with the HR-AFM. Only change the values when calibrating with a reference sample.



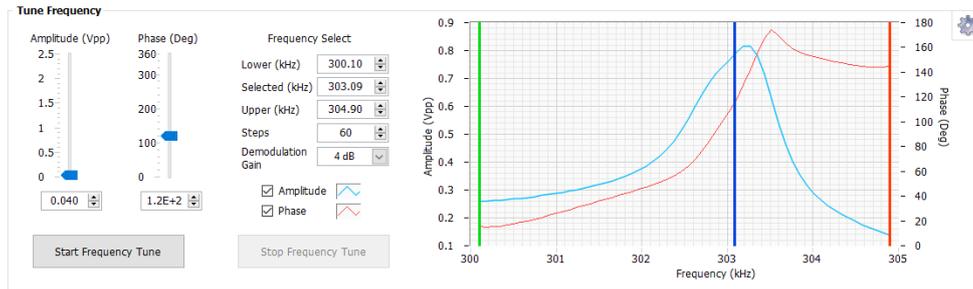
[Image 3.6: System Panel settings menu]

### 3.2.1.5 Tune Frequency

The Tune Frequency window is used for selecting the optimal conditions for producing vibrating-mode images. There are two oscilloscope windows: the upper window shows the amplitude of the probe's vibration, while the lower window shows the phase between the drive frequency and the measured frequency. The variable controls are:

1. *Amplitude* and *Phase* sliders: Adjusting the amplitude scroll bar will alter the driving amplitude of the probe's vibration. Adjusting the phase scroll bar alters the phase degree. For optimal scanning, the amplitude should not exceed 2.5V or the resonance will become saturated (see section 5.4).
2. *Lower*, *Selected*, and *Upper Frequency*: The lower, selected, and upper frequencies correspond to the green, blue, and red vertical bars in the oscilloscope windows, respectively. When trying to find the resonance peak of a new probe, it is advised to set the lower and upper frequencies to the boundaries of the frequency of the probe type, which should be found on the probe box or accompanying literature. The selected frequency should fall just to the left of the maximum of the resonance peak and on the steepest part of the slope of the phase curve, owing to attractive and repulsive forces at work on the cantilever's oscillation.
3. *Steps*: This controls the number of times the amplitude and phase are sampled as the frequency is swept. For larger ranges, this number should be larger, to avoid "Missing" the frequency peak. One step per kHz is a good starting number, but to see details of the peak, larger numbers can be used.
4. *Start* and *Stop*: These buttons can be used to start and stop frequency sweeps.
5. *Demodulation Gain*: This function adjusts the gain used in the detection of the oscillating signal from the photodetector. 4dB is a good starting value for a reference sample, but should be altered in conjunction with the drive amplitude with different probes and samples to maintain a resonance peak under 2.5V. Note

that while larger values may be necessary in some cases, maintaining this values below 13 dB usually leads to a lower noise and increased stability in the system.



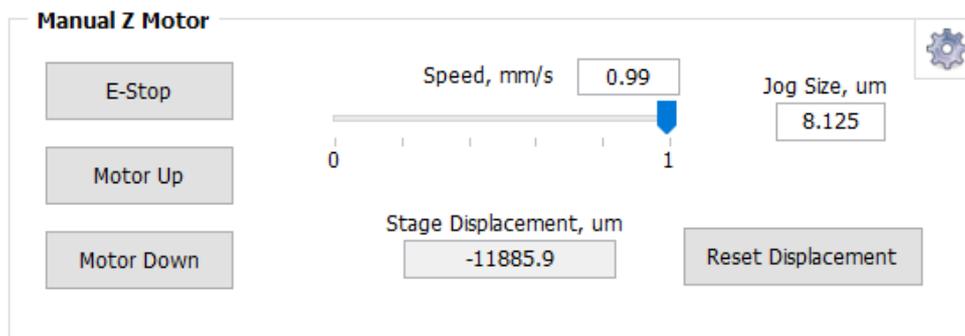
[Image 3.7: Tune Frequency Window]

### 3.2.1.6 Manual Z Motor Control

The Z motor, which raises and lowers the light lever, is activated with this window. The speed of this motion can be controlled with the speed slider. When the Up and Down buttons are held down the motor slews. The motor is jogged by touching the Up and Down buttons. The jog size can also be controlled. The stage displacement display shows how far the motor has moved. Using the “Reset Displacement” button when the probe is on the surface, allows the user a quick view of how far the probe is from the surface.



**CAUTION:** Always visually monitor the position of the Z motor. The Z motor can drive the cantilever into the sample stage and damage both the probe holder and the scanner.



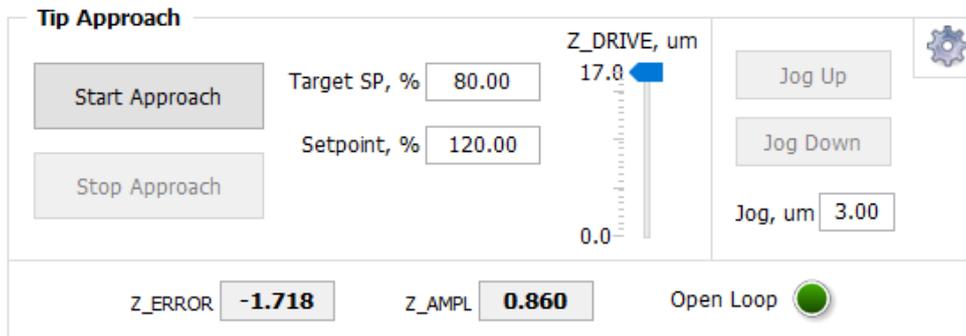
[Image 3.8: Manual Z Motor Control Panel]

### 3.2.1.7 Automated Tip Approach

The Automated Tip Approach routine is a safe method to approach the surface and put the system into feedback, without risk of damage to the probe.

A woodpecker motion is used, which results in noticeable clicking sounds from the microscope stage. This sound is from two sources: the stepper motor being energized

and the Z piezoelectric ceramic being retracted. When the system is engaged in feedback, the Open Loop indicator will light up and change to “In Feedback.”



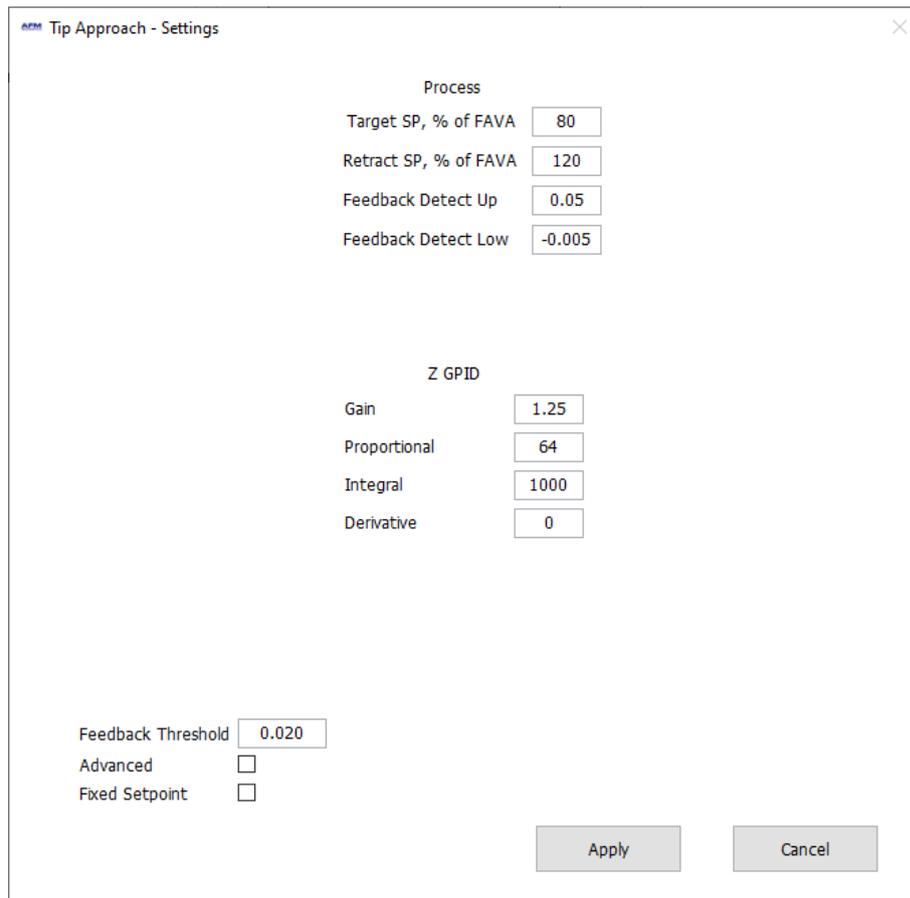
[Image:3.9 Tip approach controls]

The controls to the right of the Start Approach and Stop Approach button allow the user to alter the Target Setpoint for approach, and the current setpoint. The control to the right of this is a display, which shows the extent to which the Z piezo is extended. The movement of this control up and down gives a visual display of the progress of the approach routine.

On the far right are controls that are activated when in feedback, and allow the user to safely manipulate the extension of the Z piezo. The “Jog,  $\mu\text{m}$ ” box allows the user to choose a distance to jog the position up or down between 0.3 and 3  $\mu\text{m}$ , with the Jog Up and Jog Down buttons.

At the bottom of this panel are controls which allow the user to see the current state of the approach routine and feedback signals, for example in vibrating mode, the current vibrating amplitude is displayed, while in non-vibrating mode the Top-Bottom signal is shown.

**Settings:** The default settings window for the Tip Approach panel are shown below.



[Image 3.10: Standard tip approach settings window]

This window has various controls which can alter the way that the automated tip approach operates. In general, the user will not need to alter these values from their default values which are shown above.

Note that this settings window will have a slightly different appearance in vibrating and Non-vibrating mode. The user may want to alter these with very soft or dirty samples, or probes, or to speed up approach.

The first two values are those used to move the probe closer to the surface using the piezo, and away again, in the woodpecker method. Note: FAVA stands for Free-air Vibration Amplitude. The next two values relate to the values used to detect when the probe is in the feedback. The GPID values are those used to move the probe. Larger values will cause a faster approach. The feedback threshold value reflects how large the error signal can be for the system to consider the feedback loop to be engaged correctly. Noisy imaging conditions may require a larger value.

The Fixed Setpoint option allows a feedback where the system will always approach the Target Setpoint, irrespective of how many times the approach button is pushed, and should be used with caution. The advanced check box enables a more complex approach mechanism for very fine control of tip-sample forces during approach. The default values for this advanced routine can be seen in the window below.

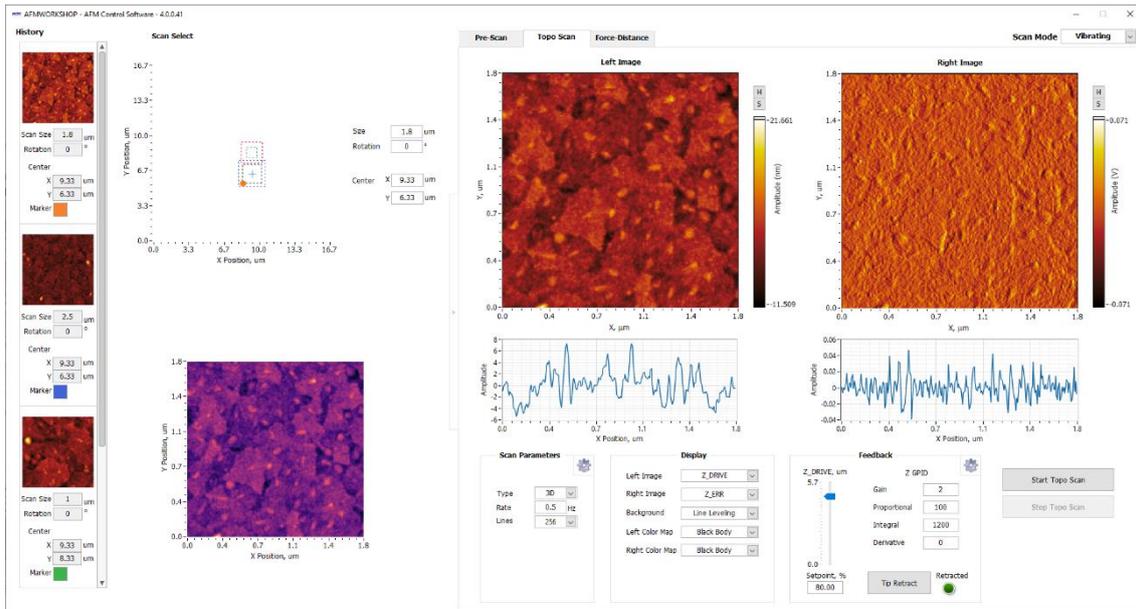
Phase 1		Phase 2		Phase 3	
Active	Yes	Active	Yes	Active	Yes
Mode	Woodpecker	Mode	Woodpecker	Mode	Woodpecker
Process		Process		Process	
Extend SP, % of FAVA	80	Extend SP, % of FAVA	80	Extend SP, % of FAVA	80
Retract SP, % of FAVA	120	Retract SP, % of FAVA	120	Retract SP, % of FAVA	120
Feedback Detect Up	0.05	Feedback Detect Up	0.05	Feedback Detect Up	0.05
Feedback Detect Low	-0.005	Feedback Detect Low	-0.005	Feedback Detect Low	-0.005
Motor Velocity, um/s	100	Motor Velocity, um/s	100	Motor Velocity, um/s	100
Z GPID		Z GPID		Z GPID	
Gain	1.25	Gain	1.25	Gain	1.25
Proportional	64	Proportional	64	Proportional	64
Integral	1000	Integral	1000	Integral	1000
Derivative	0	Derivative	0	Derivative	0
Feedback Threshold	0.020				
Advanced	<input checked="" type="checkbox"/>				
Fixed Setpoint	<input type="checkbox"/>				
<input type="button" value="Apply"/> <input type="button" value="Cancel"/>					

[Image 3.11: Advanced tip approach settings window]

The advanced setting for probe approach are shown above. The three phases allow the user to approach quickly until relatively close to the surface, then more slowly as feedback is approached, allowing fine control of tip approach forces. Reducing probe-sample forces during approach can enable the use of highly sharpened probes without damage. See chapter 6 for an in-depth explanation of high-resolution imaging.

### 3.2.2 Topo Scan Window

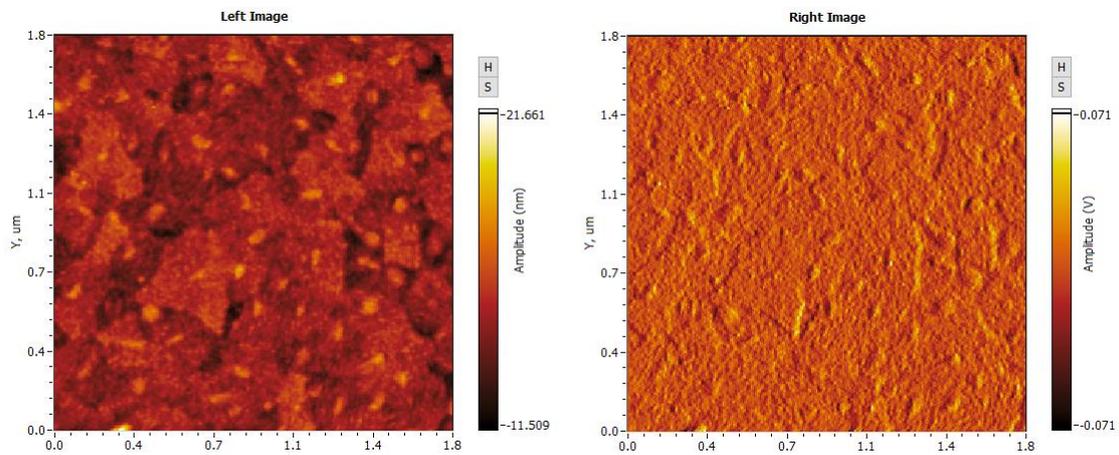
After the Pre-Scan functions are all setup, and Tip Approach completed the Topo Scan window is used for measuring AFM images.



[Image 3.12: Topo Scan Tab]

### 3.2.2.1 Image Windows

Two image channels are displayed simultaneously while scanning. The type of image data and their appearances are selected in the Display panel. As the images are displayed, there is a constant normalization of the data so that the images display the full z axis scale.<sup>9</sup>



[Image 3.13: Image windows]

The grey *H* and *S* buttons on the right side of the Image Windows allow users to open the scans in a larger window (by pushing *S*) and adjust the histogram in real time (by pushing *H*).



[Image 3.14: SH buttons]

The histogram (H) can be adjusted by moving the red and green vertical bars.

The Scan (S) button opens a larger version of the image in a new window.

### 3.2.2.2. Oscilloscope Windows

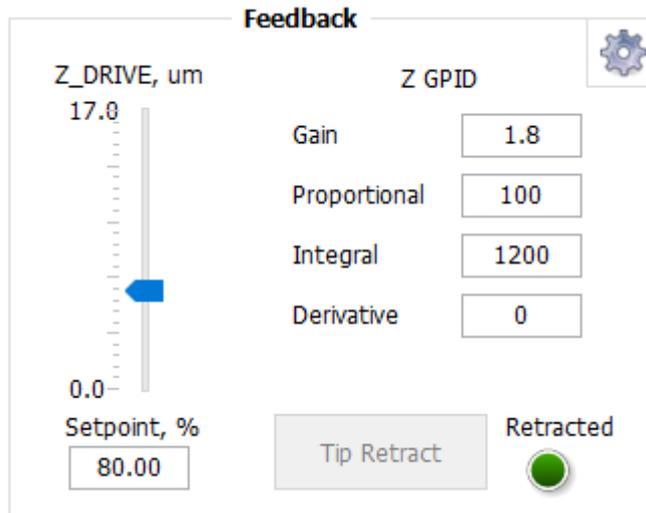
A z-height vs. x-position plot of the probe as it scans across a surface is plotted in two screens below the image scans. The data plotted is selected in the Display window. These line profiles can be useful to estimate the height of features being scanned, and to aid in feedback optimization.

### 3.2.2.3 Z Feedback

All of the parameters for maintaining feedback are provided here. The parameters are:

1. *Gain*: Scale factor for the feedback control signal.
2. *Proportional*: Scale factor for the proportional term in the PID feedback controller.
3. *Integral*: Scale factor for the integral term in the PID feedback controller.
4. *Derivative*: Scale factor for the derivative term in the PID feedback controller.
5. *Setpoint*: Parameter that controls the magnitude of interaction between the probe and surface. In the case of non-vibrating mode, larger values increase the force of interaction, while in vibrating mode, lower values indicate stronger interaction.

See Appendix G for advice on setting up GPID parameters.



[Image 3.15: Z feedback controls]



**CAUTION:** If the Z Feedback parameters are too high, the Z ceramic can begin to oscillate and potentially cause damage to the ceramic.

**Settings:** The settings controls for the Z Feedback tab include an option to change the speed at which the system moves the probe to the start of the scan, and another to control probe acceleration. In normal use, these settings will not be changed.

### 3.2.2.4 Display

There are several options for changing the appearance of the images displayed in the Scan window. They are:

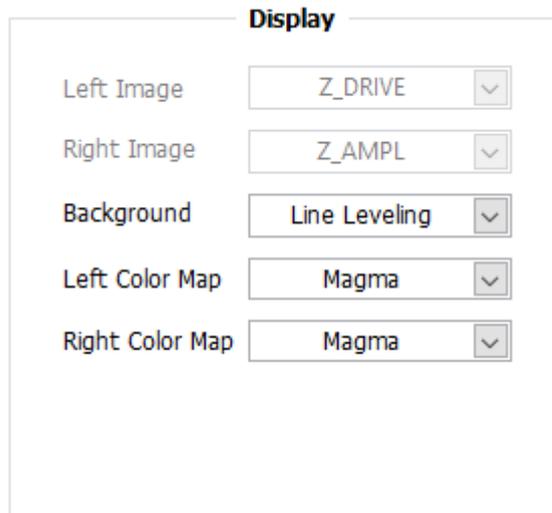
*Left Image:* The source of the image displayed in the left image window.

*Right Image:* The source of the image displayed in the right image window.

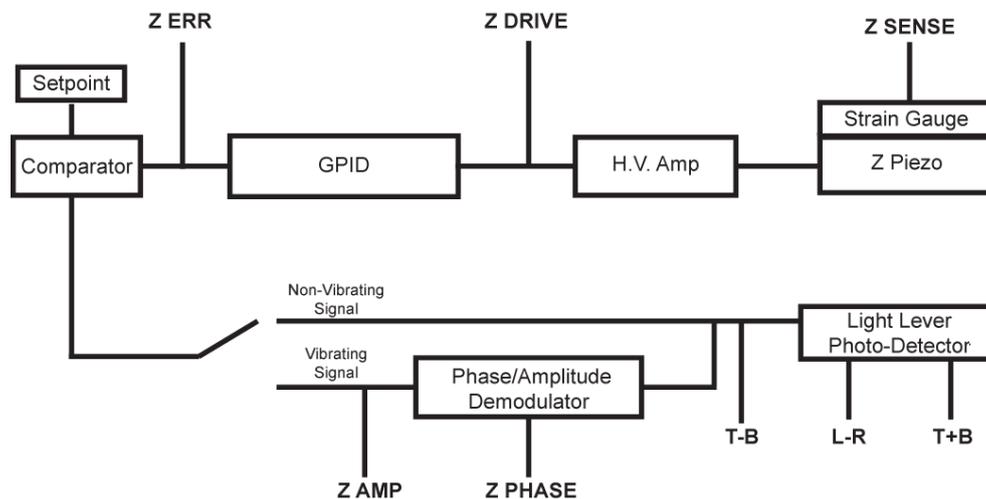
*Background:* When Line Leveling is selected, the background is subtracted from the image one line at a time. This is the best option for most images. The line leveling will not be saved in the final image file. The option No Background can be selected to view the raw data.

*Left Color Map:* This menu allows the user to select the color scheme to use for the false color maps in the Left channel.

*Right Color Map:* This menu allows the user to select the color scheme to use for the false color map in the Right channel.



[Image 3.16: Display controls]



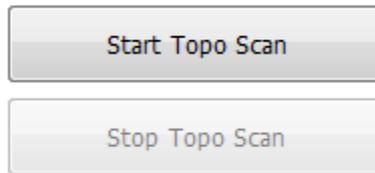
[Image 3.17: Signals block diagram]

### 3.2.2.5 Tip Retract

After scanning a sample, this function is used to move the tip away from the surface. The Tip Retract function should always be used to assure that the probe does not get damaged after scanning. Note that after a successful Tip Retract, the Retracted light will be illuminated in green, and the probe will be 50  $\mu\text{m}$  from the surface.

### 3.2.2.6 Scan

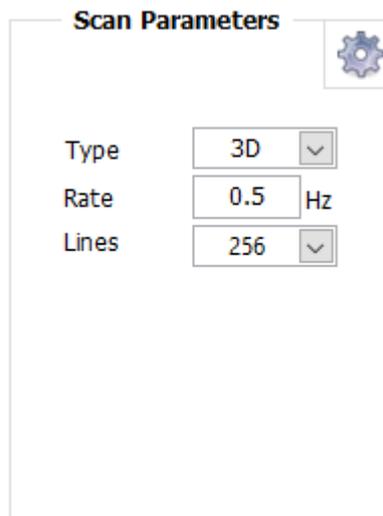
The Start Topo Scan and Stop Topo Scan buttons allow the user to start and stop scans. At the end of each scan, or if the Stop Topo Scan button is activated, the user will be prompted to save the images recorded.



[Image 3.18: Start/stop scan buttons]

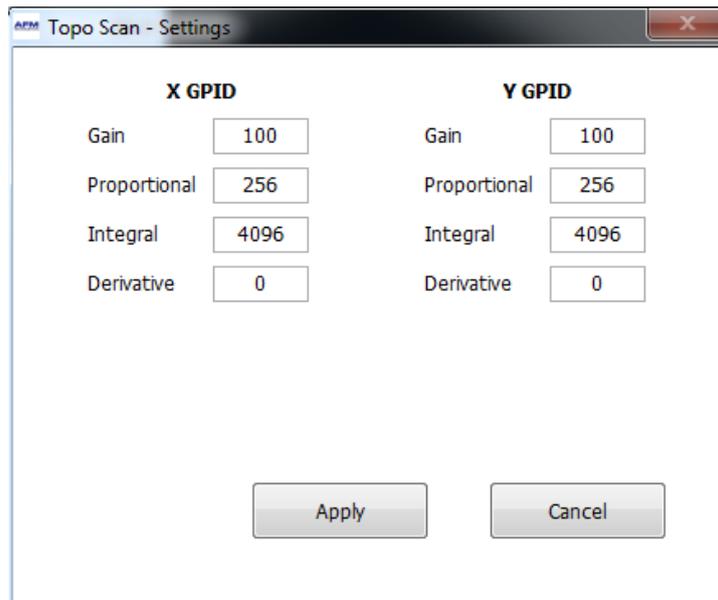
### 3.2.2.7 Scan Parameters

This panel includes the 2D/3D control which allows the user to toggle between line scanning, and full image scanning. It also allows control of scanning rate (lines per second), and the number of lines per image (the digital resolution of the image).



[Image 3.19 : Scan Parameters]

**Settings:** The parameters on the Scan parameters settings page, control the feedback between the position sensors of the HR-AFM and the X-Y piezos. In effect, they control the linearity of the resulting scan. It is recommended to only change the Gain parameter between 0 and 100 %. 0 % will give a totally non-linearized image, but enables the lowest levels of noise. 100 % gives a fully linearized scan, with moderate noise, which will be visible in small scans.



[Image 3.20 : Scan Parameters settings]

### 3.2.3 Navigation Area

The navigation area is a pop-out panel on the left of the main screen of the AFM Control software. It has two main areas, a narrow vertical History strip on the left, and the Scan Select area on the right. The Scan Select panel houses the main controls used to select the area of the sample to scan. The top square area shows the total range of the currently selected scanner. The controls on the right allow the user to select the scan range and the scan angle from 0 to 360°. Either controls can be change, by typing the desired value into the box, or by placing the cursor in the box and using the arrow up and down buttons. Below this are the “Center” controls, these control the location of the center of the area to scan. Again, they can be changed by typing, or using the cursor (the scan range must be larger than the maximum range of the scanner. In addition, this can be controlled by clicking in the scanner range, to drag the scan area center to the desired location. The blue dotted box in the Scan select area shows the currently selected scan area, with a cross in the scan center, and the orange dot shows the starting location of the probe.

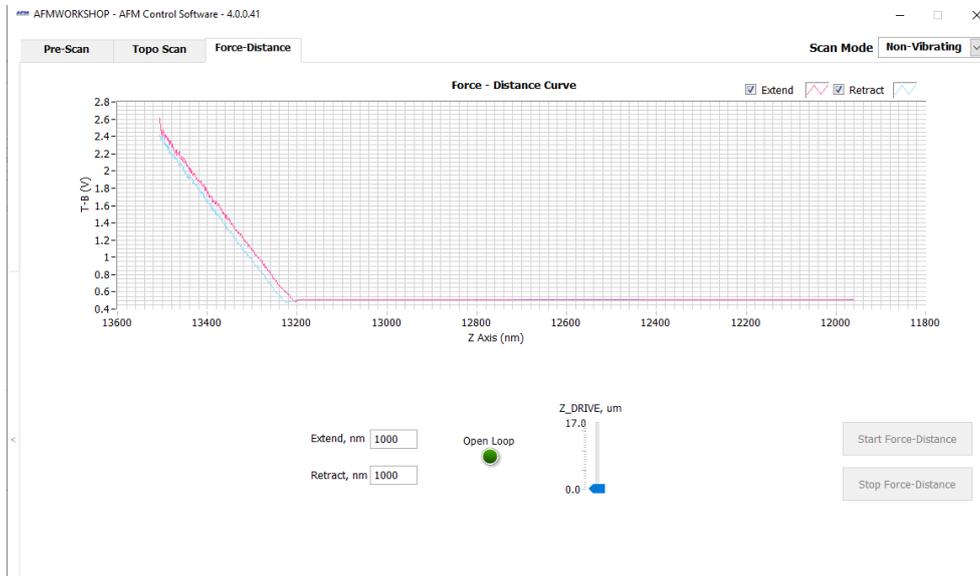
The History strip on the left of the navigation area holds the locations (center X and Y locations, scan size, and scan angle) of the previous scans made. The images saved (left image channel) are displayed in the history strip as small thumbnail images. Note that unsaved images do not appear here. Each of the thumbnails is associated with a colored marker, which shows the location of the previous scan in the scan select area. Clicking on any of the thumbnail images will load the scan size, location, scan angle and resolution back into the scan controls. This is particularly useful for zooming in and out of features within the scan range.

### 3.2.4 Force/Distance Curves

Force/distance curves are created by measuring the deflection of the cantilever as the sample is moved towards and then away from the probe at the end of the cantilever. The shape of a F/D curve depends primarily on the cantilever stiffness and material,

the nature of the sample, and the thickness of a surface contamination layer or other environment. For information about how to analyze F/D curves, see Appendix I.

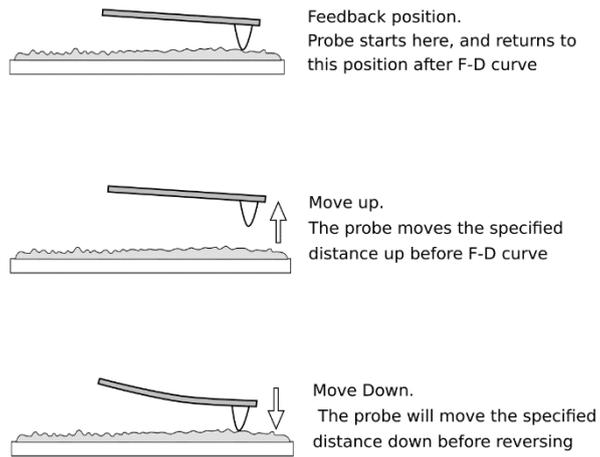
Measurement of F/D curves can be performed using the Force-Distance tab.



[Image 3.21: F-D Tab]

The AFM must be in feedback in non-vibrating mode before a F/D curve is measured. There are three important parameters that must be set before the F/D curve is measured. They are:

1. *Down*: The distance in nanometers that the sample will be moved away from the probe when the F/D curve measurement is initiated.
2. *Up*: The distance in nanometers that the sample will be moved up from the feedback position during the F/D curve. During this part of the curve, the probe is touching the sample surface.
3. *Rate*: The rate or velocity of the sample as it is moved towards and away from the probe.

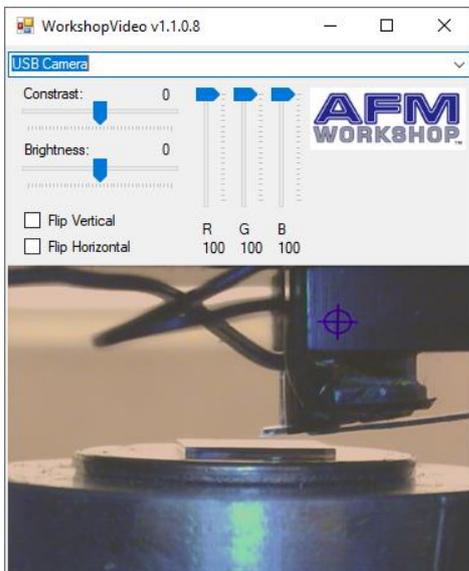


[image 3.22 probe on surface, moving down, and moving up]

**NOTE:** The advanced Force-distance software from AFMWorkshop enables considerably more detailed control over force-distance curves. The use of the advanced software is described in a separate User Guide.

### 3.3 Workshop Video Software

The Workshop Video software allows real time visualization of the probe and surface.. It is recommended to optimize the parameters for each type of sample. Before operating the video camera, it must be properly aligned; see Appendix A.



[Image 3.23: view of microscope image from side-view camera, and top-view camera]

The quality of the image viewed with the top view video microscope depends directly on the reflectivity of the sample being viewed. Therefore, it is not possible to give instructions for optimizing the video microscope images. It is recommended that operators experiment to find the best parameters.

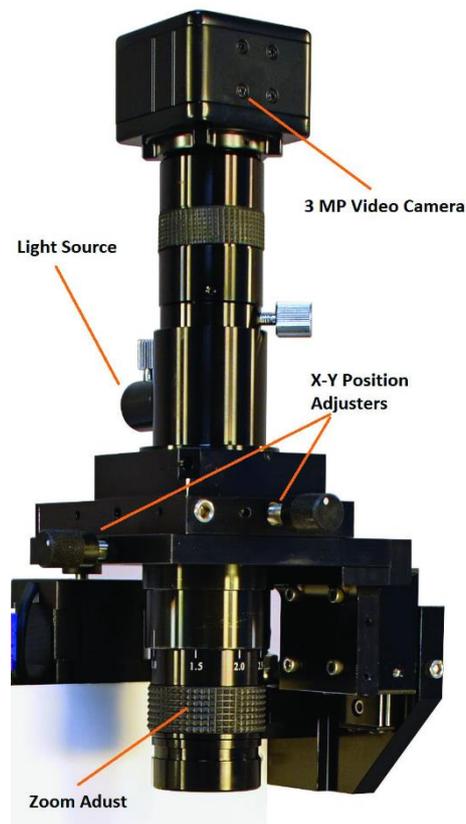
## 4. Measuring Images with the HR-AFM

Once the HR-AFM is assembled, users can expect to take a few hours to learn how to measure images. It is very important to learn how to:

1. Operate the video microscope.
2. Prepare samples.
3. Exchange probes.
4. Align the light lever force sensor.
5. Perform video-microscope-assisted tip approach.
6. Once these techniques have been mastered, measuring images is relatively simple.

### 4.1 Operating the Video Microscope

Operating the video microscope is essential for efficient operation of the HR-AFM. It allows for proper viewing of the sample, assists in manual tip approach, and is critical for laser and photo-detector alignment. It is advised to practice operating the video microscope before attempting to replace probes or measure images. The components of the video microscope are:



[Image 4.1: Video microscope controls]

1. XY Position Translator: These consist of XY position translators used for centering the video microscope window over the probe location.
2. Zoom Adjust: The zoom lens allows for viewing of a surface at high or low magnification. The light intensity and focus may need to be adjusted when making large changes with the zoom lens.
3. Focus adjust (optional): The horizontal wheel is used to focus the video microscope on the

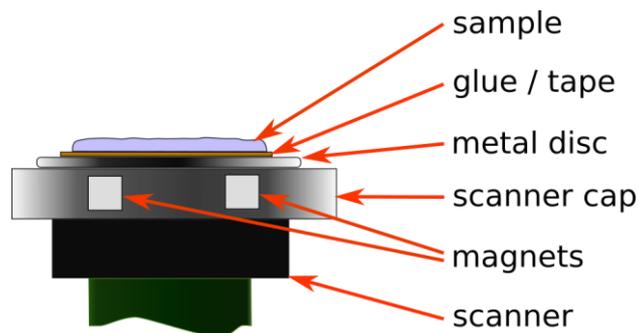
4. Light Intensity Adjust: It is often critical to adjust the intensity of the light on a sample to get the best image. This is controlled via a separate light-adjustment control box (not shown).

**NOTE:** The 1 motor and 4 motor models of the HR-AFM differ in the controls for the video microscope. The 4 motor version does not have the manual focus wheel, and uses software to control focus.

**NOTE:** since the video microscope works using reflected light, the reflective quality of the sample can affect the quality of the image obtained. With some very low-reflectivity samples, the side view camera may be required to properly visualize the probe and sample.

## 4.2 Preparing Samples

Metal disks serve as sample holders for the HR-AFM. Attach the sample to a metal disk with double-sided sticky tape or glue. Once a sample is attached to the disk, it is held onto the microscope's sample holder magnetically. It is critical that the sample be tightly bound to the sample disk in order to avoid vibrations, which degrade the image resolution.

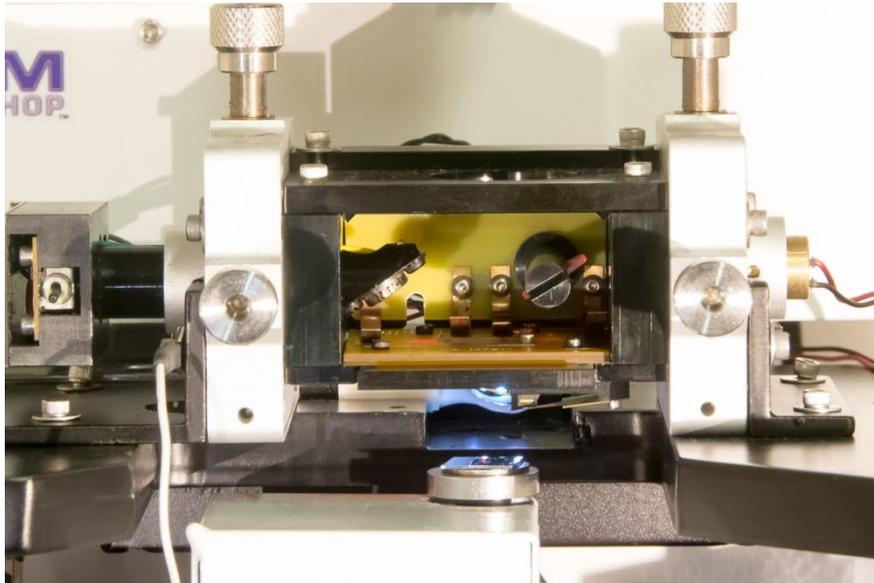


[Image 4.2 sample on scanner cartoon]

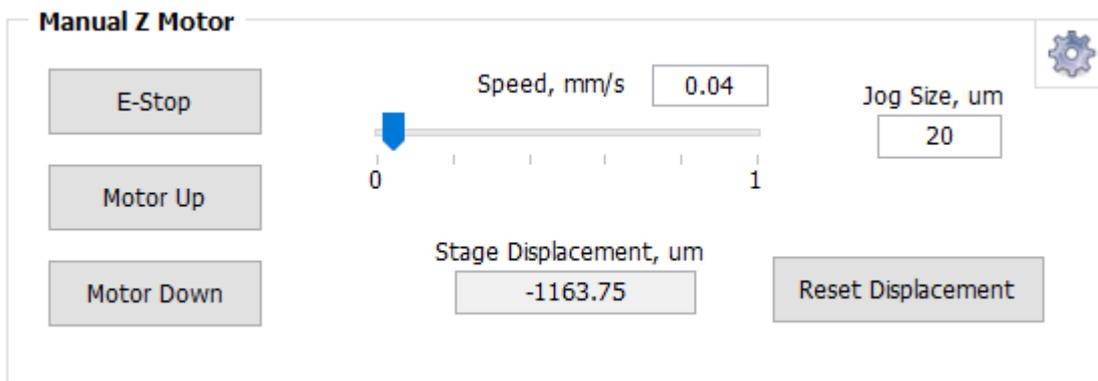
### 4.2.1 Exchanging Samples

Follow the steps below to successfully exchange samples with the HR-AFM.

1. Raise the Z motor until the probe is at least  $\frac{1}{4}$ " from the sample's surface.
2. Remove the probe holder (see section 4.3).
3. Place the sample disk on the top of the sample holder. Adjust with small, sharp tweezers until the sample is visible in the video microscope.



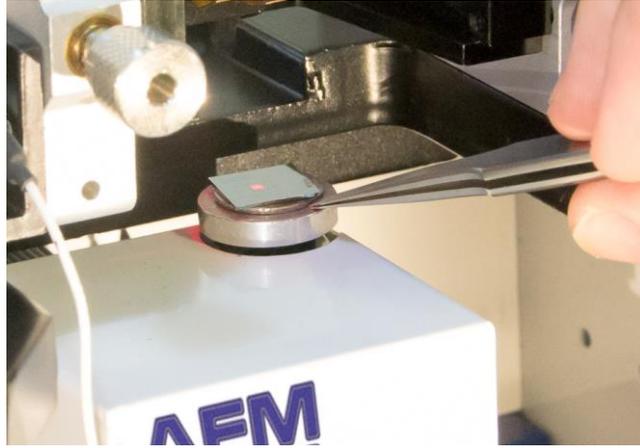
[Image 4.3: HR-AFM with raised head]



[Image 4.4: Manual Motor controls]



**CAUTION:** Do not place excessive force on the sample holder, as it could break away from the piezoelectric ceramic.



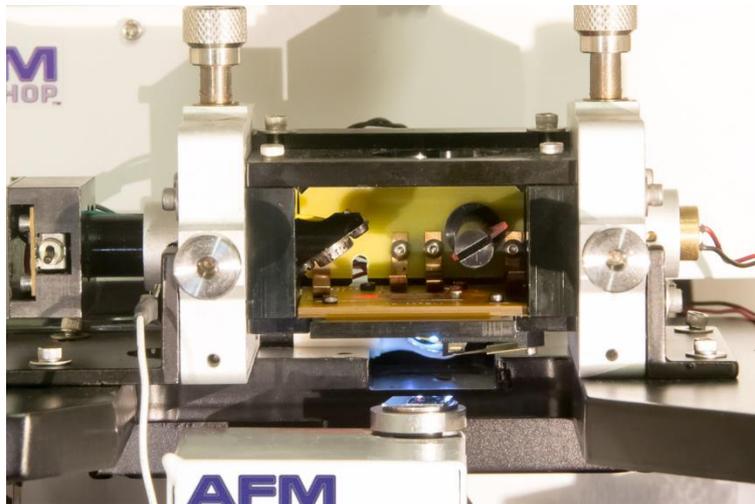
[Image 4.5: Inserting sample on scanner]

4. Align the sample using the video optics (see section 4.1).
5. Replace the probe holder with the probe tip facing the sample.

### 4.3 Exchanging Probes

When a tip becomes contaminated, damaged or broken, these steps can be followed to exchange probes quickly and efficiently.

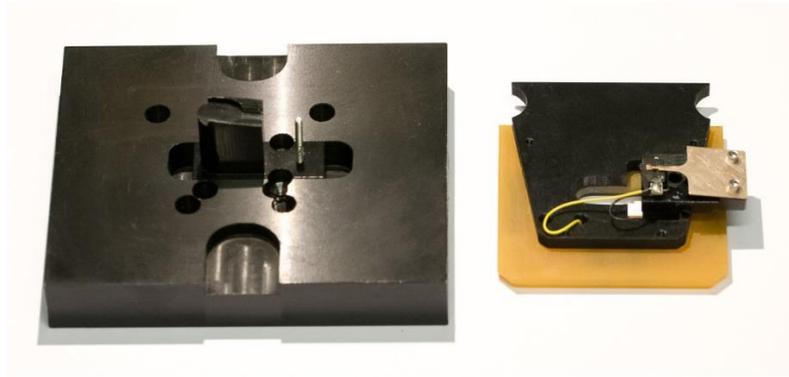
1. Raise the Z motor until the probe is at least  $\frac{1}{4}$ " from the sample's surface.



[Image 4.6 Raised head]

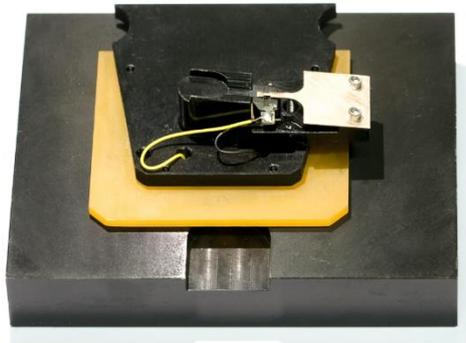
**NOTE:** Make sure the new probe is close at hand and facing in the same orientation as the probe exchange tool.

2. Remove the probe holder from the light lever.
3. Place the probe holder on the probe exchange tool with the spring facing up.

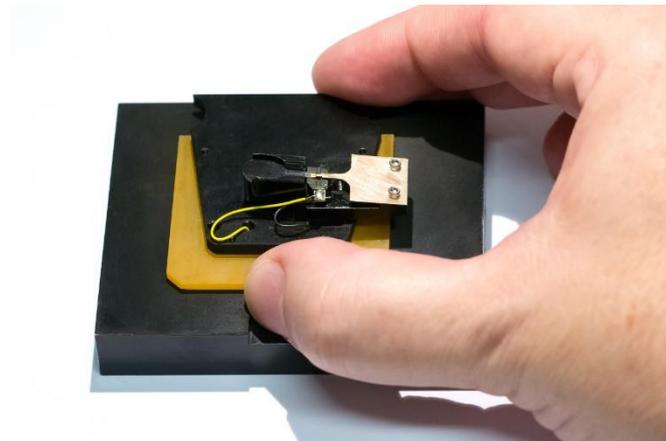


[Image 4.7: Probe exchange tool and probe holder]

4. Push down on the probe holder, such that the spring releases the probe.



[Image 4.8: Probe holder on Tip exchange tool]



[Image 4.9: Pushing down on probe holder]

5. Tap the probe exchange tool lightly on a flat surface at a 30° angle until the probe slides down through the narrow channel of the exchange tool. Gently dispose of the old probe with tweezers.

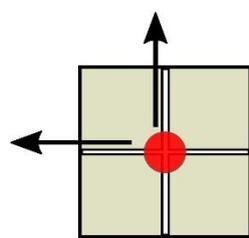
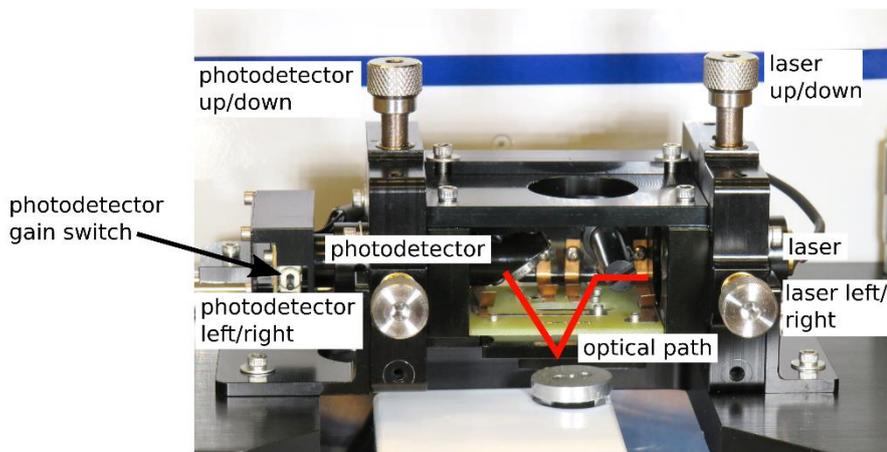
**CAUTION:** The exchange tool will not prevent expulsion of the probe if the tapping angle is too extreme.

- Using the tips of very sharp tweezers, pick up the new probe and, while pressing down on the probe holder, gently slide the probe into place. Take care not to touch the end of the chip with the cantilever.

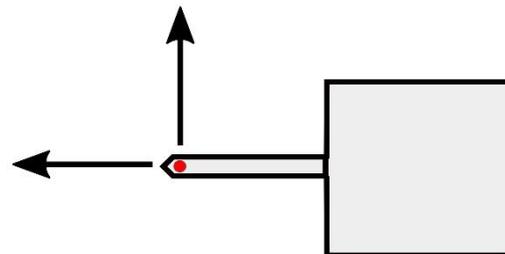
**NOTE:** If the probe holder does not sit completely level with the probe exchange mechanism, it is possible to adjust to the screws on the bottom of the exchange tool to achieve the desired height.

#### 4.4 Aligning the Light Lever Force Sensor

Aligning the light lever force sensor is greatly facilitated by understanding the optical path of the light in the sensor. The following figure shows the light path and the effect of turning the adjustment knobs on the light lever.



Motion of the laser spot when the photodetector adjust knobs are turned clockwise



Motion of the laser spot when the laser adjust knobs are turned clockwise

[Image 4.10: Light lever scheme]

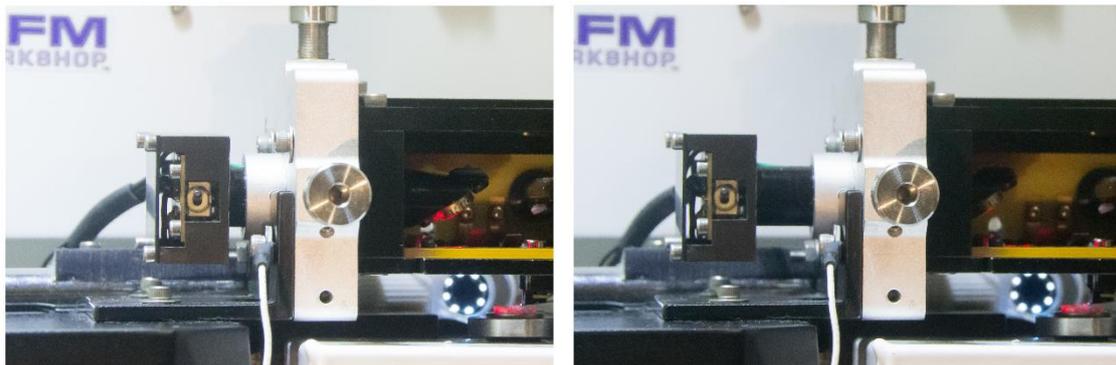
##### 4.4.1 Tips for Aligning the Light Lever Force Sensor

- Bringing the cantilever within a millimeter of the sample's surface can help the user see the laser on the sample.
- After replacing a cantilever, the laser and photo-detector adjustments are usually less than a single turn.

3. The adjustment screws may have backlash; sometimes the motion is not perfectly linear.
4. If the cantilever does not align within a few minutes, try another cantilever.
5. The detector has two modes: high and low gain. High gain allows more laser light through the detector than low gain. High gain is commonly used for most scanning purposes because it has a better signal to noise ratio.
6. When in vibrating mode, if the resonance peak appears inverted at the top, reduce the amplitude slider. If further adjustments are necessary, switch the detector gain from high to low.
7. If the Beam Position is not aligned within a few turns of the adjustment knobs, the photo-detector can be manually adjusted by manually gripping the photo detector, and sliding it a small distance in or out of the light lever head. Repositioning the detector so that the Top + Bottom signal is maximized while the beam position is mostly centered. Normally some small adjustment with the photodetector screws will be required after this operation

**NOTE:** In normal use, there is not requirement to slide the photodetector in or out of the light lever head. This may become necessary in two situations:

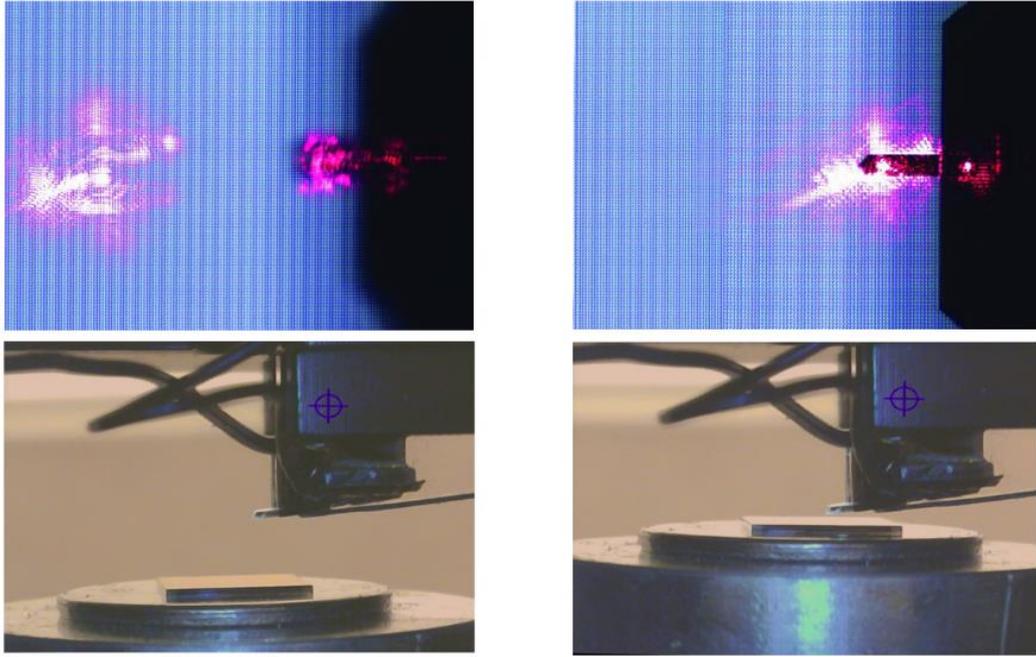
1. When changing from a very long to very short type of probe, or vice-versa.
2. When changing from air-scanning to scanning in liquid or vice-versa.



[Image 4.11: photo detector pushed in and out]

#### 4.5 Optically-Assisted Tip Approach

Tip approach with the HR-AFM is achieved using a woodpecker algorithm activated in the Pre-Scan window. To save time during tip approach, move the probe as close to the surface as possible before activating the Automatic Tip Approach function. The video microscope and side-view can prove very helpful for manual probe approach. Below are video microscope images (top and side-views) of a probe as it moves toward a standard reference sample surface.

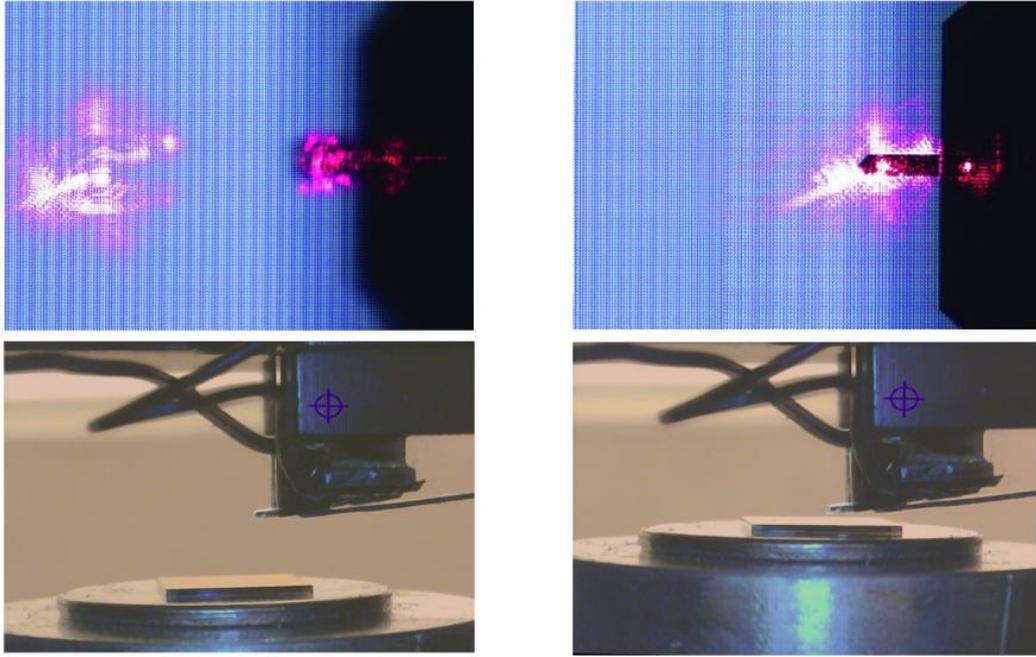


[Image 4.12: Probe far from surface (left), and probe close to surface (right), top view (above) and side-view (below)]

Probe far from surface (left), and probe close to surface (right), top view (above) and side-view (below)

#### 4.5.1 Performing Optically-Assisted Tip Approach

1. Focus the video microscope on the sample surface.
2. Carefully lower the Z motor with the Manual Z Motor Control, until the cantilever is visible in the video microscope image. Make sure the Jog Size has been set to a low step size (such as 5-10  $\mu\text{m}$ ) and the Z motor speed is set to 0.01 mm/sec in order to avoid crashing the probe into the sample.
3. Slowly jog the probe down until it is only slightly out of focus.
4. As shown below, the side-view camera on the HR\_AFM can also be useful to bring the probe close to the surface.



[Image 4.13: tip far from and close to surface]



**CAUTION:** If the tip touches the surface before the feedback is activated, the tip may be damaged or even broken.

5. The system is now ready for Automated Tip Approach.

## 4.6 Contact Mode Imaging

These instructions assume the AFM Control Software is properly loaded and working, and that the material presented in section 4 has been reviewed.

### 4.6.1 Pre-Scan Tab

1. Follow the instructions in section 4.2. and place a sample in the instrument.
2. Follow the instructions in section 4.3. and place a contact mode probe in the instrument. See Appendix E for probe recommendations.
3. Select Non-Vibrating in the Scan Mode window.
4. Focus the video optical microscope on the AFM probe.
5. Follow the instructions in section 4.4. to align the laser on the probe. Make sure the Top – Bottom signal is between 0.3 and 0.8 for contact-mode imaging.
6. Focus the video microscope on the sample surface.
7. If this is the first scan since opening the AFM Control Software, click on Range Check in the System window.

**NOTE:** The probe must NOT be in contact with the sample surface during Range Check. The piezoelectric ceramics will move to measure the usable range, which may cause damage to the tip.

8. Use the Manual Z Motor Control to move the light lever towards the sample (see section 4.5.).

9. Initiate Automated Tip Approach. Wait for the green light to indicate that feedback was achieved.
10. If the Z Drive indicator is above the center of its range, use the Jog Down button to move the Z Drive indicator towards the middle. You may need to change the Jog size, depending on the scanner in use (a good size is 3  $\mu\text{m}$  for a 17  $\mu\text{m}$  scanner, or 1  $\mu\text{m}$  for a 7  $\mu\text{m}$  range scanner). A few jogs may be required to reach the center of the range.
11. If the Z Drive indicator is below the center of its range, use the Jog Up button to move motor up by small increments until the indicator is at the center of the Z range.
12. Once true feedback has been achieved, open the Topo Scan tab at the top of the AFM Workshop window.



[Image 4.14: Pre-scan Tab in non-vibrating mode with numbers]

#### 4.6.2 Topo Scan Tab

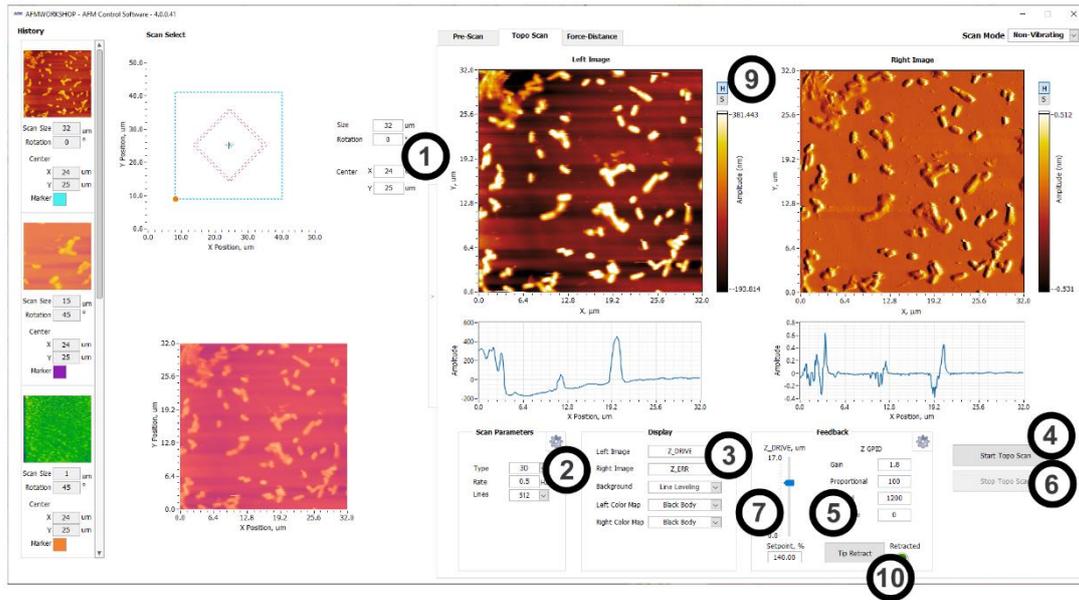
1. Select the parameters controlling the area to be scanned; these include the scan size, the center position in x and y, and the scan rotation. Samples may be scanned between 0° or 360° by adjusting the Rotation parameter.

NOTE: A scan rate of 0.5 Hz and 256 scan lines will achieve a basic low resolution scan.

2. Select the Scan Rate (scanning speed, in lines per second) and the number of lines (digital resolution of the image).
3. Select the desired images to be displayed in the Display window. The Display window also allows for control of leveling. Some display options for contact (non-vibrating) mode scanning include:
  - Z-Drive, the change in voltage through the z-piezoelectric ceramic, shows the sample's topography. It is common to set Z-Drive to the Left Image.
  - Z-Error, the error signal, is used to help optimize the Z Feedback parameters. When initially setting up a scan, it is common to use Z-ERR as the Right Image.
  - T-B signal is similar to Z-Error, and is sometimes used as the Right Image
  - L-R, lateral force, detects cantilever torsion as the tip applies constant force along the surface. This mode is unique to contact mode scanning, and can only be properly used when scanning at 90°.
4. Begin a scan by pressing the Start button in the Scan window.
5. While scanning, optimize the GPID and setpoint parameters (see Appendix G).

**NOTE:** When the GPID parameters are optimized, the Z piezoelectric ceramic will more closely follow the probe tip as it scans the surface. When tip movement is minimized the Z error signal is decreased. **Increasing** the setpoint by small increments (50-100 mV) can sharpen the image by pushing the tip harder on the surface.

6. Once the parameters are optimized, stop the scan by pressing the Stop button. Save the image if desired.
7. Before beginning a new scan, check to see if Z Drive is still near the center of its range. If not, see sections 4.6.10 - 11.
8. Begin a new scan. When the scan is completed, the user will be prompted to save the image.
9. If desired, the user can open a larger window of both the Left and Right Images by selecting the S button to the right of the scan window. The H button allows the user to alter the histogram of the scans, which can help to visualize small features.
10. When finished scanning, press Tip Retract. This function gently releases the probe from the sample surface.



[Image 4.15: topo scan tab with numbers]

## 4.7 Vibrating Mode Imaging

These instructions assume the AFM-View software is properly loaded and working, and that the material presented in section 4 has been reviewed.

### 4.7.1 Pre-Scan Tab

1. Follow the instructions in section 4.2 and place a sample in the instrument.
2. Follow the instructions in section 4.3 and place a vibrating mode probe in the instrument. See Appendix E for probe recommendations.
3. Select Vibrating in the Scan Mode window.
4. Focus the video optical microscope on the AFM probe.
5. Follow the instructions in section 4.4 to align the laser on the probe. Make sure the Top – Bottom signal is close to 0.0 for vibrating mode imaging.

**NOTE:** The probe must NOT be in contact with the sample surface during Range Check. The piezoelectric ceramics will move to measure the usable range, which may cause damage to the tip.

6. If this is the first scan since opening the AFM-View software, initiate the Range Check function.
7. Select the frequency under the Tune Frequency window.
  - a) Set the Lower limit to the lowest frequency on the probe box.
  - b) Set the Higher limit to the highest frequency on the probe box.
  - c) Set the Selected value to any frequency between the two limits. If the selected frequency exceeds either limit, the software will automatically change the values.
  - d) Set the number of Steps (50-200) and the Demodulation Gain (1-10 dB).
  - e) Click Start Frequency Tune to sweep of the selected range.
  - f) Move the green and red cursors toward the resonance peak to decrease the range, and place the blue cursor corresponding with the selected frequency

- 80% up the left side of the resonance peak. At this point, the number of Steps can be created to 50, to speed up the process, if desired.
- g) Repeat steps (d) and (e) until a resonance peak no greater than 2.5V is observed. Optimal imaging can depend on the value used, with 1.0V often used as a starting point. If the amplitude of the resonance peak exceeds 2.5V, adjust the driving amplitude of vibration by altering the Amplitude scroll bar, or by directly typing a value into the box below.
  - h) For phase mode imaging, the resonance peak must correspond with the slope of the phase curve. To alter the phase degree, adjust the Phase (deg) scroll bar accordingly. If phase imaging will not be performed, unchecking the Phase option will remove the phase plot, making visualization of the amplitude plot easier.
8. Focus the video microscope on the sample surface.
  9. Use the Manual Z Motor Control to move the light lever towards the sample (see section 4.5.).
  10. Initiate Automated Tip Approach. Wait for the green light to indicate that feedback was achieved.
  11. If the Z Drive indicator is above the center of its range, use the Jog Down button to move the Z Drive indicator towards the middle. You may need to change the Jog size, depending on the scanner in use (a good size is 3  $\mu\text{m}$  for a 17  $\mu\text{m}$  scanner, or 1  $\mu\text{m}$  for a 7  $\mu\text{m}$  range scanner). A few jogs may be required to reach the center of the range.
  12. If the Z Drive indicator is below the center of its range, use the Jog Up button to move motor up by small increments until the indicator is at the center of the Z range.
  13. Once true feedback has been achieved, open the Topo Scan tab at the top of the AFM Workshop window.



[Image 4.16: Pre-scan window in vibrating mode with numbers.]

#### 4.7.2 Topo Scan Tab

1. Select scan parameters such as Scan Size, Scan Rate and the number of lines. Samples may be scanned between 0° and 360° by adjusting the Rotation parameter.

**NOTE:** A scan rate of 0.5 Hz and 256 scan lines will achieve a basic low resolution scan.

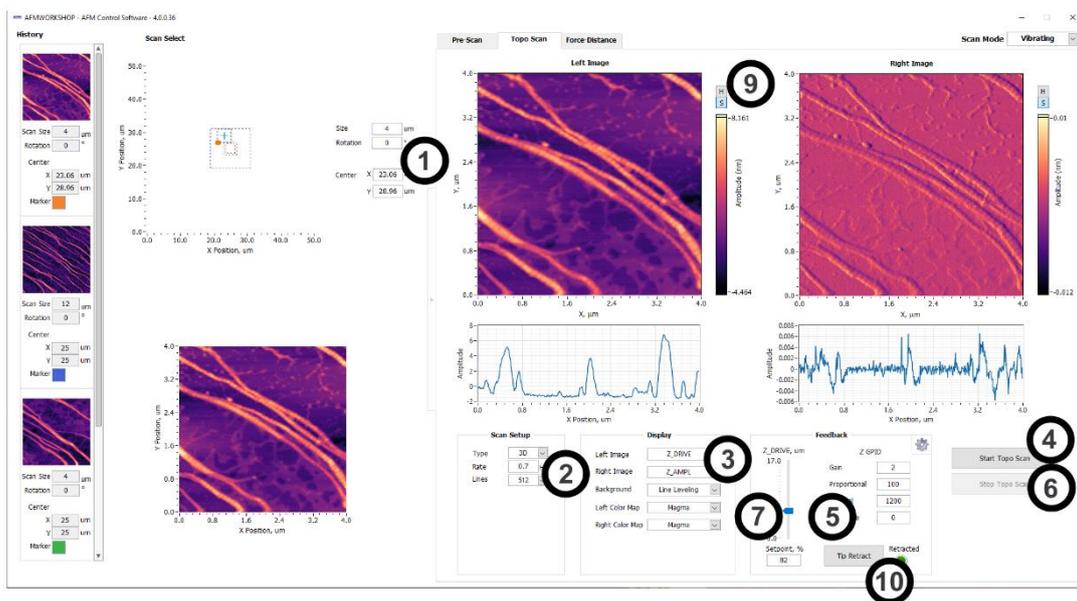
2. Select the desired images to be displayed in the Display window. The Display window also allows for control of line leveling. Some display options for vibrating mode scanning include:
  - Z-Drive, the change in voltage through the z-piezoelectric ceramic, shows the sample's topography. It is common to set Z-Drive to the Left Image.
  - Z-Error, the error signal, is used to help optimize the Z Feedback parameters. When initially setting up a scan, it is common to use Z-ERR as the Right Image.
  - Z\_AMPL, the Amplitude signal, which is similar to the Z-Error signal. It is quite common to use the Amplitude signal as the right image.
  - Z-Phase, phase imaging mode, uses the differences in the probe's oscillation to form image contrast. The phase shifts when the probe comes into contact with areas of the sample surface with differing stiffness, adhesion, etc. Thus phase

imaging mode is optimal for composite samples, those with varying surface densities and characteristics.

3. Begin a scan by pressing the Start Topo Scan button in the Scan window.
4. While scanning, optimize the GPID and setpoint parameters (instructions in Appendix G).

**NOTE:** When the GPID parameters are optimized, the Z piezoelectric ceramic will more closely follow the probe tip as it scans the surface. When tip movement is minimized the Z error signal is decreased. **Decreasing** the setpoint by small increments (10-100 mV) can sharpen the image by pushing the tip harder onto the surface.

5. Once the parameters are optimized, stop the scan by pressing the Stop button.
6. Before beginning a new scan, check to see if Z Drive is still at the center of its range. If not, see sections 4.6.10 - 11.
7. Begin a new scan. When the scan is completed, the user will be prompted to save the image.
8. If desired, the user can open a larger window of both the Left and Right Images by selecting the S button to the right of the scan window. The H button allows the user to alter the histogram of the scans, making it easier to visualize small features.
9. When finished scanning, press Tip Retract. This function gently releases the probe from the sample surface.



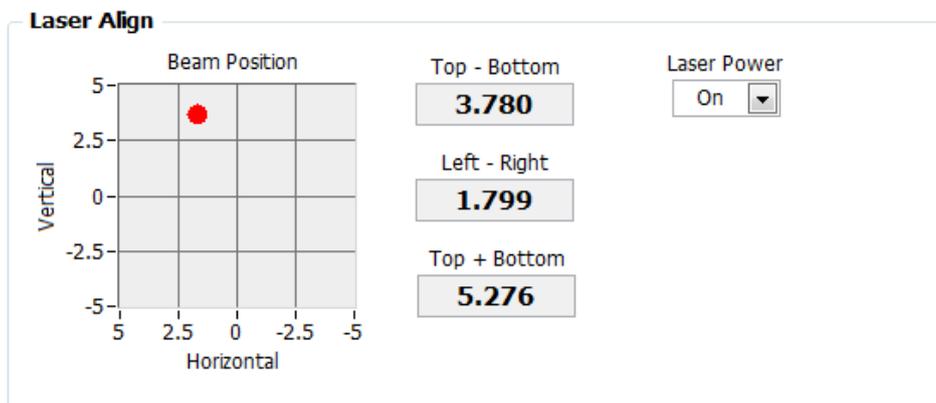
[Image 4.17: Topo scan window in vibrating mode with numbers]

## 5. Troubleshooting – What Can Go Wrong When Measuring Images

Although the operation of the HR-AFM is quite simple, some problems can occur while imaging samples. The following sections detail some of the most common problems that may occur. It is important to keep these potential problems in mind when operating the HR-AFM.

### 5.1 Saturation of the Photo-Detector

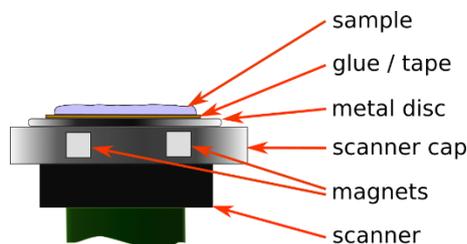
Highly-reflective cantilevers can sometimes pass too much laser light to the photo-detector. When this occurs, the light lever force sensor will not work properly. When there is too much light, the Top + Bottom signal in the Laser Align window displays a value greater than 3. This problem can often be solved by flipping the gain switch on the photo-detector down to low gain (see section 4.4 for the location of the gain switch).



[image 5.1: saturated photodetector]

### 5.2 Sample Not Mounted Correctly

It is essential that the sample is rigidly mounted on the sample holding plate. If the sample is not mounted rigidly, it can be difficult to get into feedback, and images appear to have excessive vibrations. Equally, the sample must not move, or have loose debris that can shift during imaging.

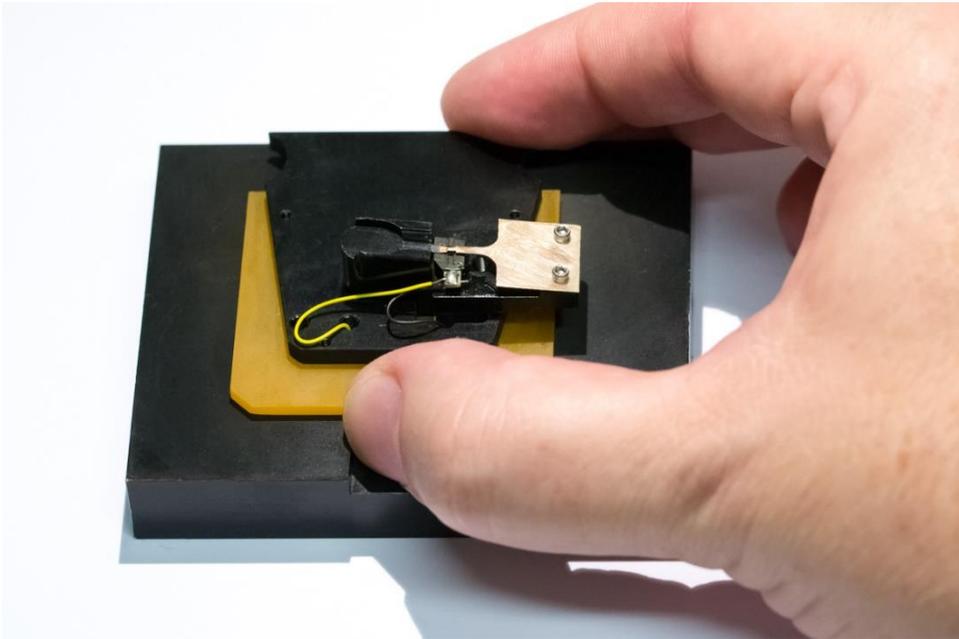


[image 5.2: mounted sample]

### 5.3 Probe Not Seated Correctly

If the probe is seated correctly, it will be in essentially the same place before and after the probe is removed from the AFM. If it is too difficult to align the light lever after a probe exchange, examine the probe holder closely to make sure that the probe is

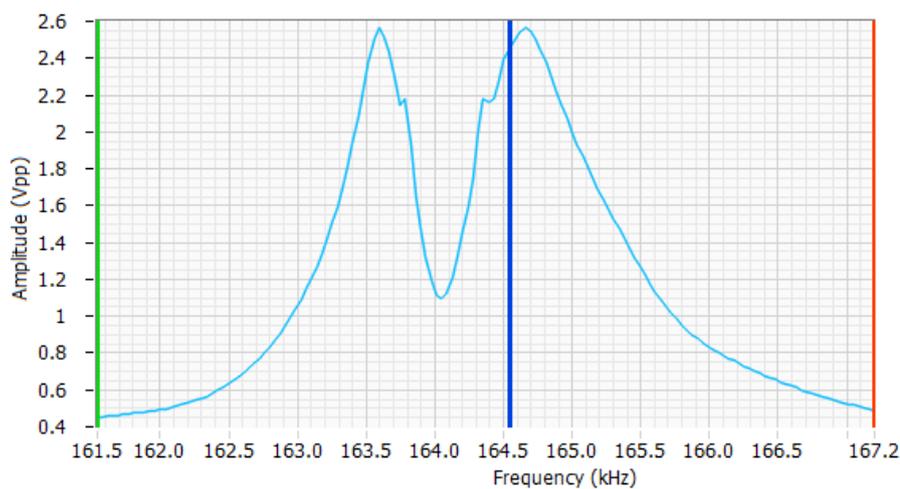
seated correctly. Improper probe alignment may cause resonance saturation. If the probe holder is dirty, it may also cause problems.



[image5.3: probe in probe holder]

#### 5.4 Resonance Saturated

When scanning the frequency of the cantilever produces a double-peaked resonance, as shown on the right, decrease the driving amplitude of the probe by lowering the Amplitude scroll bar until the resonance peak sits below 2.5V. If the resonance is still saturated, the probe may have to be re-seated.

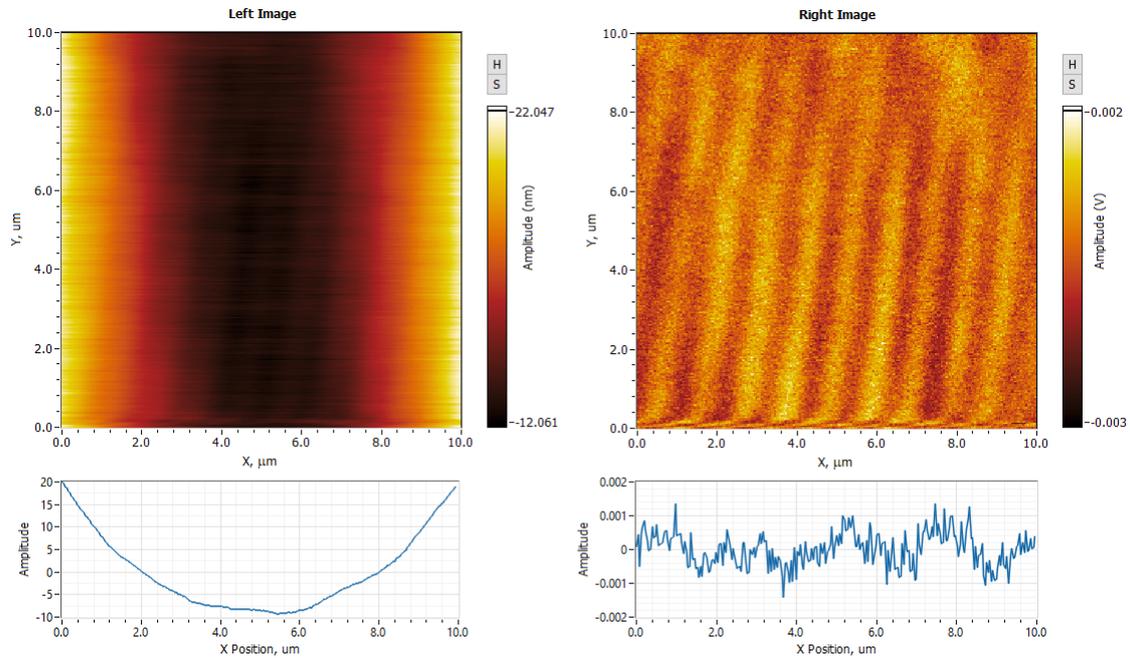


[Image5.4: saturated resonance]

#### 5.5 False Feedback

In vibrating mode, the probe can go into false feedback, resulting in wispy, featureless images. False feedback is created when the probe is caught up in the surface contamination layer. This can be corrected by reducing setpoint, activating the tip

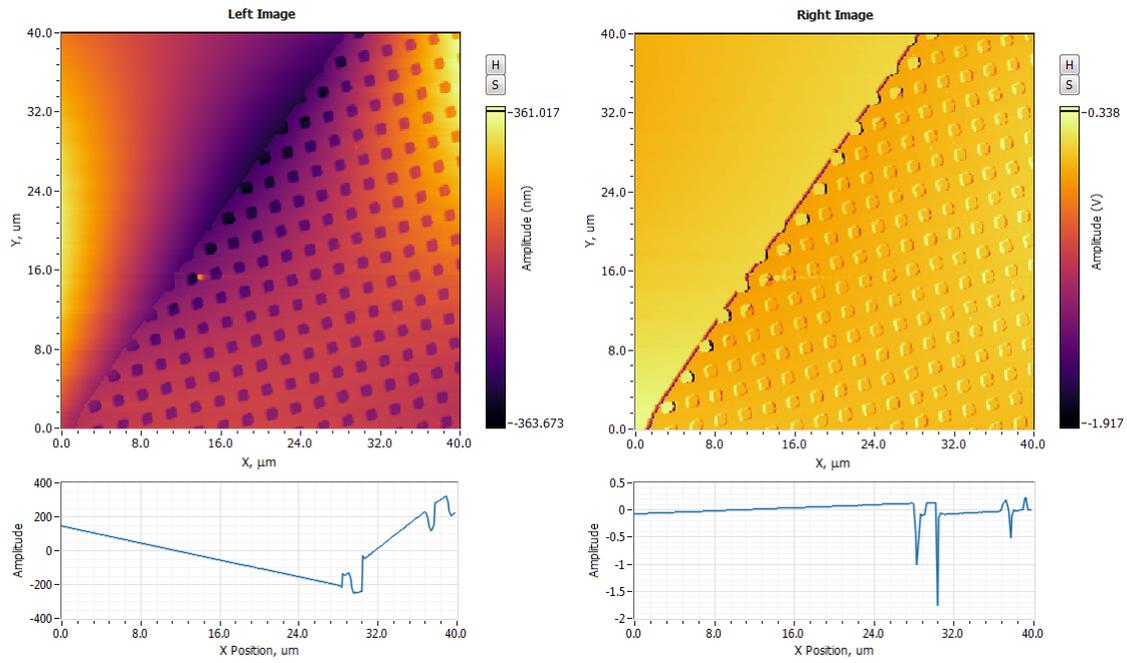
approach function a second or third time, as well as by utilizing the advanced tip approach functions to jog the tip toward the surface (see section 6).



[Image 5.5: false feedback]

## 5.6 Scanner at End of Z Range

When the Z ceramic expands or retracts to the end of its range while scanning, the image will suddenly lose contrast and appear flat. To solve this problem, relocate the sample so that the area being scanned lies directly over the center of the z piezo, (the screw hole of the sample holder). Next, redo tip approach so that the Z ceramic is in the center of its range, or use the Jog Up and Jog Down buttons (see section 4.6. j-k).



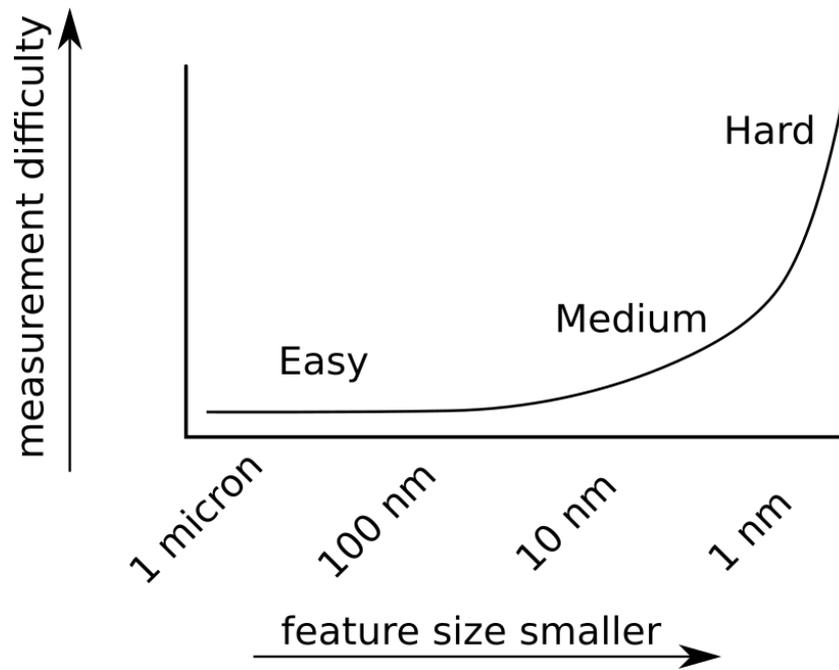
[image5.6: Scanner out of range]

## 5.7 Defective Probe

In the event that none of the above problems exist and it is not possible to get an image, the probe may be defective. Images obtained with defective probe sometimes show repeating patterns.

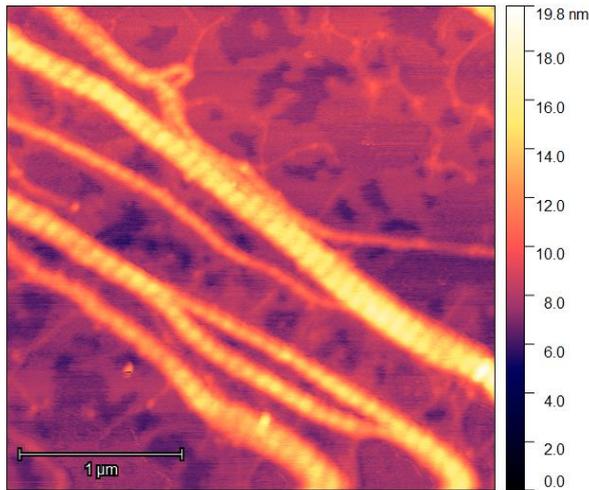
## 6. High Resolution Imaging

High resolution images are measurable with the HR-AFM. However, as with any AFM, obtaining those images requires that the AFM be optimally set up and that the operator knows the critical parameters for measuring high resolution images. The graph below illustrates the difficulty in measuring images as a function of the desired dimensions.

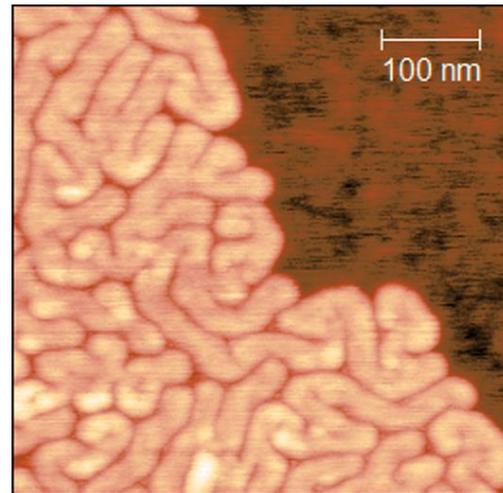


[Image 6.1: Graph]

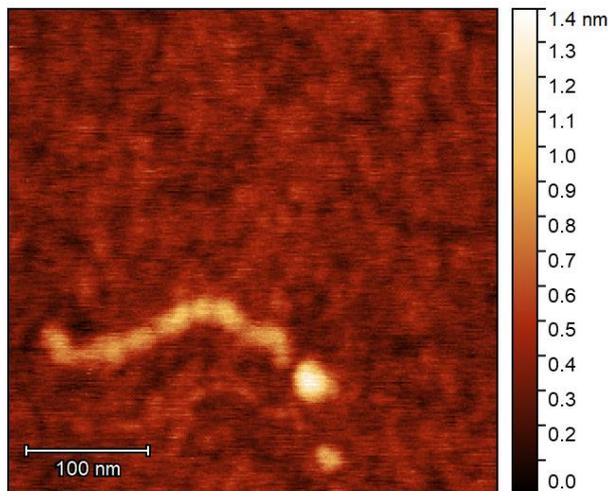
Examples of high resolution images obtained with the HR-AFM include:



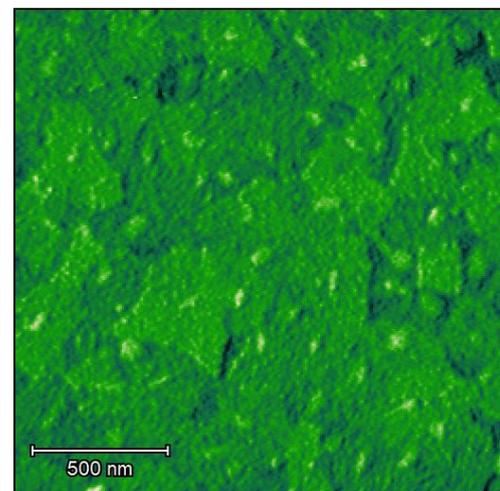
Collagen fibrils on mica



500x500x6 nm image of fluoropolymers on silicon



200 nm long DNA molecule



1800x1800x35nm image of Indium Tin Oxide

[Image 6.2: some high res images]

There are three critical requirements for measuring high resolution images with the HR-AFM:

1. The instrument must not be influenced by structural and acoustic vibrations.

For high resolution imaging, the AFM must be placed in an environment with minimal vibrations. Typically, both a vibration table as well as an acoustic enclosure is required. Sometimes, extremely sensitive measurements must be made in the middle of the night when there are minimal structural vibrations. Measuring the noise floor of the AFM establishes the Z resolution of the AFM. Measuring images of structures with 0.3 nm in vertical dimension requires a Z noise floor below 0.1 nm. See Appendix D to learn how to measure the noise floor of the HR-AFM.

2. The Electronics Gain must be properly set up.

The scanner used for the measurements must be capable of resolving the desired features. The resolution of the scanner depends on the electronic noise in the control electronics and the mechanical gain of the piezoelectric ceramics in the scanner.

In the HR-AFM the electronic noise can be reduced by reducing the Gain on the high voltage amplifiers that drive the scanner piezoelectric ceramics. In the advanced software on the HR-AFM, this is done by using the “Higher” or “Highest” Resolution Modes. Alternatively, use a scanner with a small mechanical gain. The 15 micron scanner has a mechanical gain of 1:1 and the 50 micron scanner has an XY mechanical gain of 5:1. The table below shows the electronic levels that can be expected at different electronic gains on the HR-AFM,

<b>Theoretical Resolution For 50 and 15 μm Scanners*</b>				
<b>Z HV Gain</b>	<b>50 μm Scanner</b>		<b>15 μm Scanner</b>	
	<b>Z Range (nm)</b>	<b>Z Noise (nm)</b>	<b>Z Range (nm)</b>	<b>Z Noise (nm)</b>
<b>15</b>	17,000	0.85	8,500	0.43
<b>14</b>	15,867	0.79	7,937	0.41
<b>13</b>	14,733	0.74	7,371	0.39
<b>12</b>	13,600	0.68	6,804	0.36
<b>11</b>	12,467	0.62	6,237	0.33
<b>10</b>	11,333	0.57	5,670	0.30
<b>9</b>	10,200	0.51	5,103	0.27
<b>8</b>	9,067	0.45	4,536	0.24
<b>7</b>	7,933	0.40	3,969	0.21
<b>6</b>	6,800	0.34	2,402	0.18
<b>5</b>	5,667	0.28	2,835	0.15
<b>4</b>	4,533	0.23	2,268	0.12
<b>3</b>	3,400	0.17	1,791	0.09
<b>2</b>	2,267	0.11	1,135	0.06
<b>1</b>	1,133	0.06	567	0.03

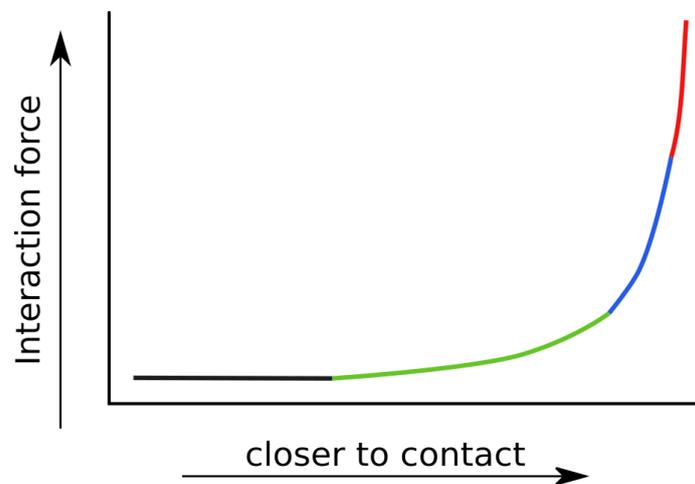
AFM Workshop's 100 μm scanner has the same Z characteristics as the 50 μm scanner.

As lower Z high voltage gains are used, the maximum Z range decreases. Therefore, the step size of the Z motor must be reduced when lower Z gains are used. This will ensure that the probe does not crash into the sample surface on tip approach. The software automatically adjusts the tip approach motor jog for changes in the Z HV Gain.

3. Tip approach must be made such that the tip is not broken.

Preserving a sharp tip for imaging requires making a perfect tip approach. This means that the probe must not crash into the surface and break the tip off. The best method for preserving the tip in probe approach is to bring the tip to the surface and engage in a false feedback. Then, very gently bring the tip into feedback such that the tip is interacting with the surface.

The following graph shows the interaction forces of the probe with the surface.



[Image 6.3: Force vs distance]

There are 4 regions of interaction forces between the tip and surface:

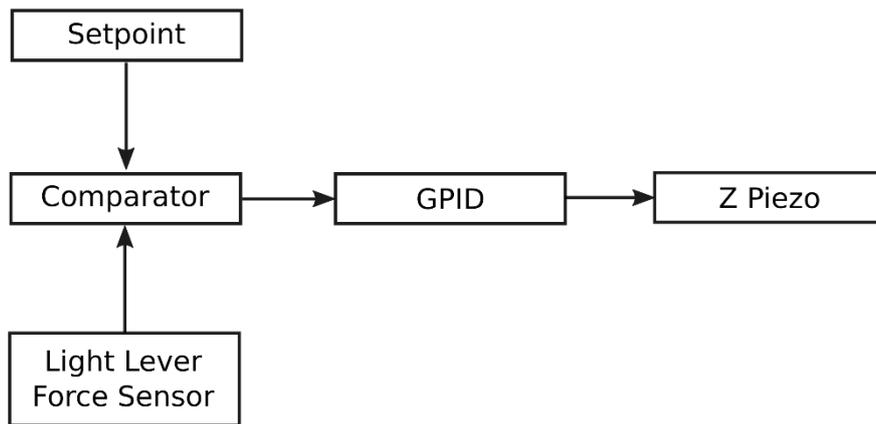
- BLACK - No interaction force
- GREEN - Acoustic interaction forces (also known as compression forces)
- BLUE - Interaction of the tip with the surface contamination
- RED - Direct interaction between the probe and the surface.

These interaction forces will cause the vibrating cantilever to decrease in its amplitude as it passes through each of these interaction regimes.

The method for getting into perfect feedback is to stop the tip approach in the green regime, and then slowly bring the probe into the direct interaction regime. This is achieved by setting the feedback threshold low such that the tip approach stops when the probe is interacting with the surface contamination (In vibrating mode, Target Setpoint around 90%). Then the probe can be slowly moved towards the surface using the advanced tip approach functions. The user will know when the tip has entered the

direct interaction regime when the yellow Z Drive slider no longer moves as the sample is pulled toward the tip via small setpoint changes.

The AFM control software is designed to make a careful probe approach under automated control. This is achieved with the Advanced Tip Approach window under the Pre-Scan tab in conjunction with the Tip Approach parameters in the **settings** window. The figure below shows how the signal from the light lever is compared to the feedback, and used to control the movement of the z piezo.



The AFM Control Software uses a “woodpecker” approach mechanism. This is a controlled stepwise movement towards the surface as explained below.

Tip Approach Sequence in Vibrating Mode			
Step	Action	Result	Tip Approach Parameter
1	Measure vibration amplitude of the probe away from the surface	Amplitude used for initial tip approach. Set point is raised and lowered around this value which causes the sample to move up and down	Z Amplitude value in the Automated Tip Approach Window
2	Raise set point	Z piezo moves toward the probe	Extend Factor
3	Check for feedback	If in feedback, end tip approach.	Feedback Detect
4	Lower set point	Moves sample away from probe	Retract Delta

5	Activate stepper motor	Moves probe towards sample	Tip Approach Motor Jog
6	Repeat steps 2-5	Feedback achieved with full Z Drive range	

It is advantageous to move the sample up and down towards the probe at a slow rate to avoid crashing the probe into the surface. The rate at which the sample moves up and down depends on:

- The magnitude of the initial Z Amplitude
- Tip approach GPID parameters
- Target Setpoint
- Retract Setpoint

The initial value for the setpoint when feedback is established is the Z Amplitude.

The image shows two parts of the control interface. On the left is the 'Tip Approach' control panel, and on the right is the 'Tip Approach - Settings' panel.

**Tip Approach Control Panel:**

- Buttons: Start Approach, Stop Approach
- Target SP, %: 80.00
- Setpoint, %: 120.00
- Z\_DRIVE, um: 17.0 (with a slider bar from 0.0 to 17.0)
- Jog Up, Jog Down buttons
- Jog, um: 3.00
- Real-time data: Z\_ERROR: -1.718, Z\_AMPL: 0.860
- Status: Open Loop (indicated by a green dot)

**Tip Approach - Settings Panel:**

- Process:**
  - Target SP, % of FAVA: 80
  - Retract SP, % of FAVA: 120
  - Feedback Detect Up: 0.05
  - Feedback Detect Low: -0.005
- Z GPID:**
  - Gain: 1.25
  - Proportional: 64
  - Integral: 1000
  - Derivative: 0

[Image 6.5: approach controls and settings]

## Appendix A: Setting Up the HR-AFM

### A.1 Selecting a Location

Structural vibrations from a building's floor and acoustic vibrations (noise) can greatly reduce the resolution of an AFM. Ideally, the AFM would be placed in a vibration-free environment, but this is typically not possible. A few tips:

- Set the AFM up in the basement or lower floor near a load-bearing wall.
- Avoid locating the AFM in the path of fans or other sources of wind turbulence.
- Avoid locations where there are elevators, a lot of foot traffic, or freeways.
- If you must place the AFM in an undesirable location, consider purchasing a vibration table and/or acoustic enclosure.

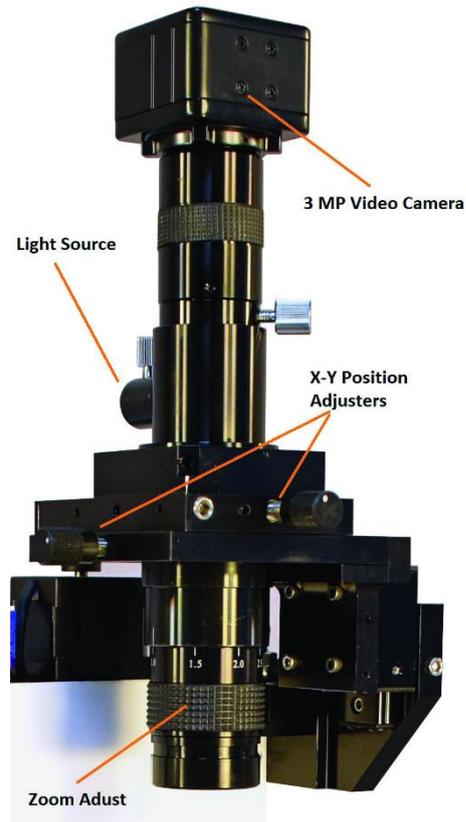
A simple test to see if a location has a problem is to fill a coffee cup with coffee and set it in the potential location. If there are ripples in the coffee's surface, there are going to be problems.

### A.2 Setting Up the Optical Microscope

The video optical microscope is essential for efficient operation of the HR-AFM. After setting up the microscope, spend some time learning to operate it by viewing various samples.

#### 1. Optical Microscope

- a) Install the zoom tube and CMOS camera, The USB cable receptor at the top of the CMOS camera should be facing forward
- b) Attach the USB cable to the camera and computer
- c) Attach the LED to the zoom tube, and connect the light power cable from the LED to the light intensity adjuster
- d) Remove the black cap from the bottom of the zoom tube



[Figure A1: Optical video microscope]

2. Test the microscope and verify its operation.

- a) Launch the Workshop Video software (see section 3.3).
- b) Turn on the light and adjust the intensity to its mid-range.
- c) Adjust the height of the zoom tube so that the bottom of the tube is 115 mm from the surface of the scanner.
- d) Place a coin or other metal object under the objective. The light should be visible on the object.
- e) On the zoom tube, turn the zoom adjustment counter clockwise all the way, thus reducing the magnification.
- f) At this time, an image should be visible on the computer screen; use the focus adjust on the video microscope to sharpen the image.
- g) Increase the magnification with the zoom tube adjuster and increase the light intensity as needed.



[Image A2: zoom images with ruler]

### 3. Setting up the side-view camera

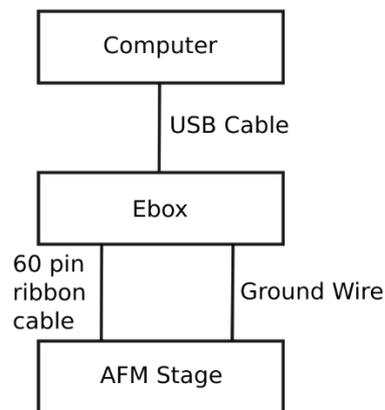
- (a) Plug the USB Cable of the side view camera in to the video microscope PC
- (b) Launch the side view camera software
- (c) Adjust the light using the rotating knob attached to the cable of the side view camera.
- (d) Optionally, place the white background holder on the front side of the AFM stage to improve the contrast on the image
- (e) The image can be easily zoomed into the probe location using the mouse wheel

#### A.3 Connecting the HR-AFM to the Cables

Connect the USB cable between the computer and the Ebox.

Connect the 60-pin ribbon cable between the Ebox and the HR-AFM stage.

Connect the grounding wire between the Ebox and the HR-AFM stage.



[Image A3: Wires block diagrams]



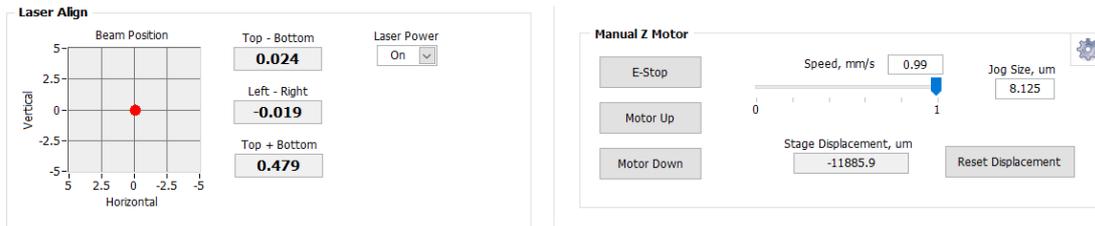
**WARNING:** Failure to connect the ground wire can cause damage to the stage and/or Ebox electronic circuits.



**WARNING:** Always turn off the master power to the Ebox before plugging or unplugging any of the cables on the HR-AFM.

## A4 System Check

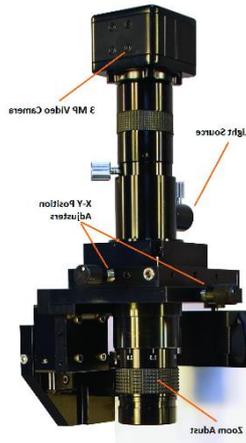
1. Turn on the computer and the Ebox.
2. Launch the AFM Control Software. The laser should now be visible.
3. Using the Manual Z Motor Control in the Pre-Scan tab, move the Z motor a few steps.
4. If the Z motor does not respond and the Top-Bottom numbers in the Laser Align section of the Pre-Scan tab are not changing, the Ebox is not communicating with the computer. In this case, cycle power to the Ebox and restart the AFM View software.



[Image A4: Images of Z motor and laser controls]

## A5. Aligning the Video and HR-AFM Microscopes

1. Raise the zoom tube on the optical microscope so that the light lever can fit under the bottom of the zoom tube.
2. Move the micrometers on the video microscope XY translators to their central position. Move the micrometers on the HR-AFM stage to their central position.
3. Place the HR-AFM stage under the zoom tube such that the hole in the light lever is directly below the zoom tube optical objective.
4. Adjust the height of the zoom tube so that the bottom of the tube is about 115 mm from the top of the AFM sample holder. Move the stage by hand and adjust the optical focus.
5. Place a probe in the probe holder (see section 4.3.) and place it into the light lever.
6. With the Z motor control, move the probe such that is about  $\frac{1}{2}$  in. above the sample plate.
7. With the focus adjustment knob, raise the zoom tube approximately  $\frac{1}{2}$  in.
8. Slowly move the stage by hand until the probe is viewed in the video microscope window. As required, adjust the focus, the lighting, and the position of the stage until the probe is in the center of the optical view

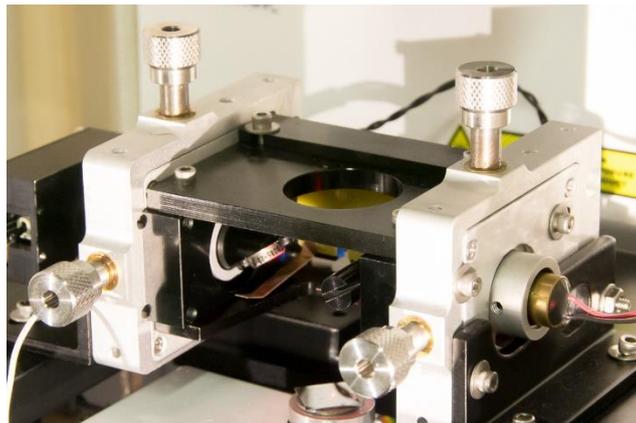


[Image A5 video microscope]

### A6. First Alignment of the Light Lever

This procedure assumes that the light lever was correctly aligned at the factory or by an authorized agent.

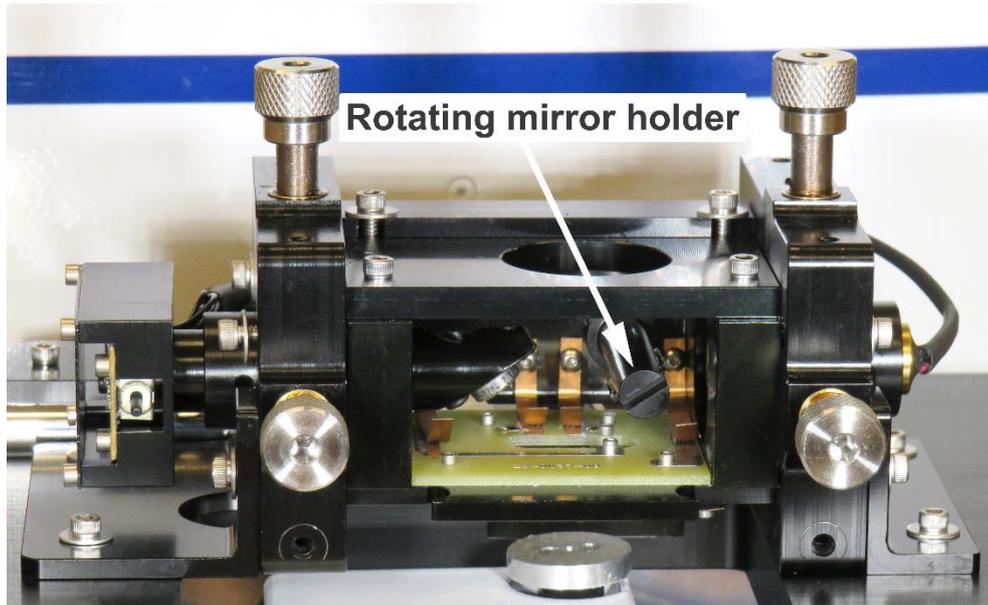
1. Place the sample disk on the sample holder.
2. Place the probe holder in the light lever with a contact or vibrating mode probe installed.
3. Adjust the optical microscope until the probe/substrate can be viewed on screen.
4. Center the laser and detector translation mechanisms with thumb screws.



[Image A6: thumb screws]

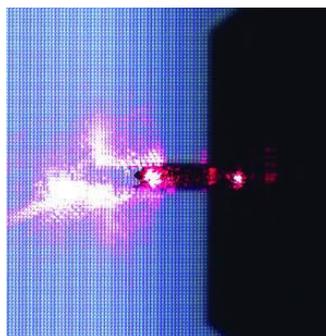
5. Turn on the laser.
6. Use a flathead screwdriver to rotate the mirror until the laser spot is on the back side of the cantilever.
7. Lower the Z motor until the probe is just above the sample disk.

8. Rotate the mirror to move the laser light until it is visible on the probe.



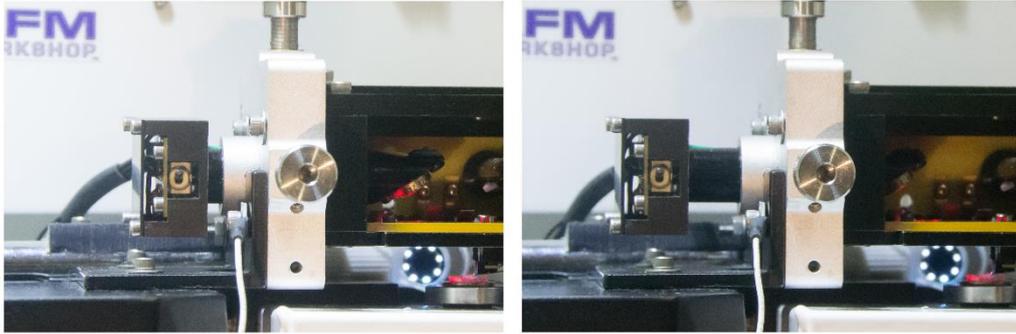
[Image A7: rotating mirror]

9. Loosen the set screws that hold the laser.
10. Slide the laser in or out until it is focused.
11. Tighten the set screws to hold the laser in place.
12. Move the laser spot out to the edge of the cantilever.



[Image A8 laser on lever]

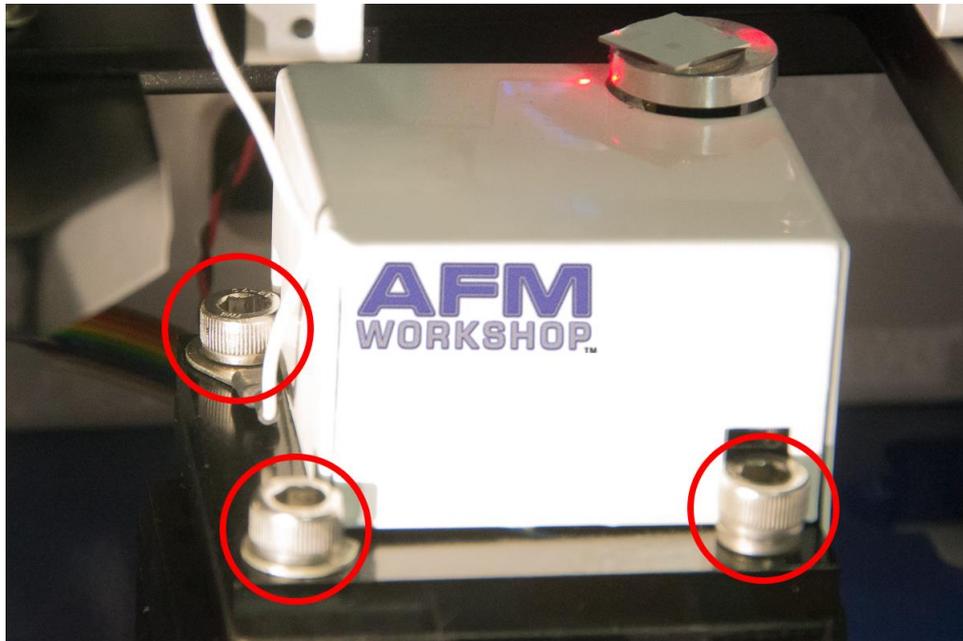
13. Release the set screws that hold the photo-detector in place.
14. Move the photo-detector in and out until the T-B signal is zero. Tighten the set screws.
15. Move the L-R signal to zero by adjusting the detector translation thumb screws.



[Image A9: photodetector]

### A7. Installing the XYZ Piezoelectric Scanner

1. If not already turned off, turn off the Ebox electronics.



[Image A10: Scanner hold down screws]

2. Place the scanner on the HR-AFM XY translator and locate the ribbon cable that plugs into the scanner.
3. Plug in the ribbon cable. The connector is located on the back of the scanner.
4. Screw in the three socket screws that hold the scanner onto the XY translator.



**CAUTION:** Do not twist the ribbon cable. Doing so will damage the HR-AFM electronics.

## A8. Leveling Sample Holder

The leveling sample holder allows the user to simply adjust the tilt of the sample being scanned. To use, simply place the leveling sample holder on the regular sample holder on the scanner, aligning the magnets. To adjust, tighten or loosen the three adjustment screws on the leveling sample holder until proper leveling is achieved. Ensuring that the sample is level will help to reduce problems such as reaching the end of the scanner's z range.



[Image A11: Levelling sample holder]

## A9. Grounding the HR-AFM

There are several grounding options for the HR-AFM. The green ground wire should be used to connect the stage with the Ebox. The probe is grounded by a grounding connector, which should be attached to the Modes/IO connection on the back of the stage. A wire can be added to the ground connector to receive signals from the probe.



[Image A12: grounding plug]

## Appendix B: HR-AFM Data Files

HR-AFM data files are stored in a text format. At the beginning of the data file are stored many of the parameters used for the scan. It can be useful to check these in order to reproduce scanning conditions. The parameters can be easily observed by opening the file in a text editor, such as notepad, or using the “metadata Browser” function in Gwyddion.

```
AFM_scan_2020-07-20_14.57.13, 8um, 256 pixels, Left.wsf - Notepad
File Edit Format View Help
AFM_scan_2020-07-20_14.57.13, 8um, 256 pixels, Left.wsf

Pixels in X: 256
Lines in Y: 256
X Range: 8.000
Y Range: 8.000
Z-Drive Calibration: 0.5600
Z-Sense Calibration: 1.0000
X Calibration: 0.6700
Y Calibration: 0.5500
X Offset: 20.667
Y Offset: 19.000
Rotation(°): 0
Scan Rate(Hz): 0.50
Scan Type: Vibrating
Vpp: 0.020
Phase: 0.00
Demod Gain : 10dB
Selected (kHz) : 303.05
Display Type: Z_DRIVE
Samples/Pixel: 20
XY HV Gain: 8
Z HV Gain: 12
Z Sensor Gain: 640
X GPID: 0.00000000 %, 256, 4096, 0
Y GPID: 0.00000000 %, 256, 4096, 0
Z GPID: 2.1974, 110, 1300, 0
Setpoint: 482

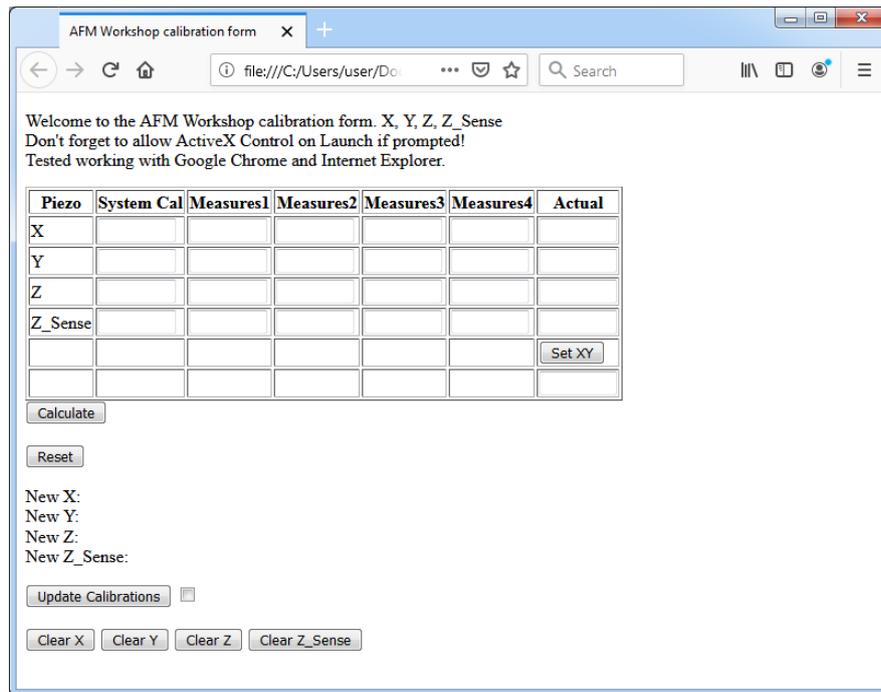
-4781.1706 -4790.4856 -4801.0179 -4804.5902 -4791.6230 -4784.7490
-4783.0336 -4786.3845 -4794.9678 -4805.8260 -4815.0180 -4793.7135
-4774.7885 -4766.1375 -4763.9610 -4766.5618 -4774.6778 -4786.4276
-4797.5133 -4790.3687 -4783.5808 -4785.6713 -4794.3284 -4792.8835
```

[Image B1: screenshot showing metadata]

## Appendix C: Calibration

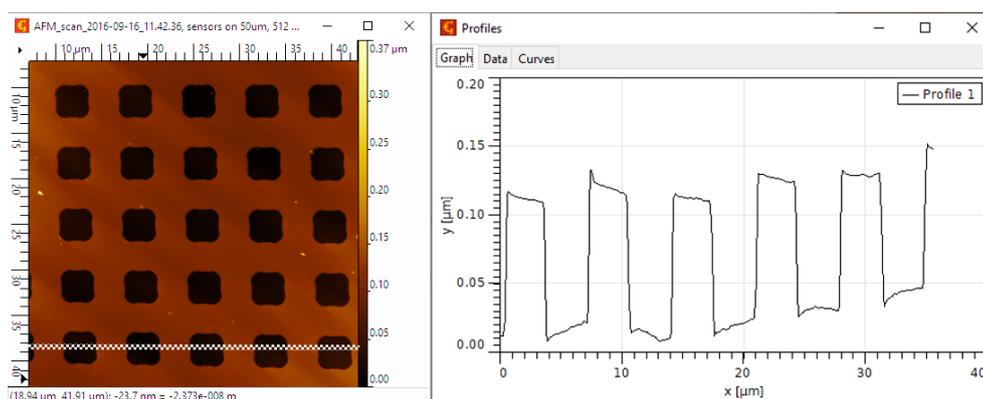
Calibration of the HR-AFM requires scanning a standard sample that has known X, Y and Z dimensions. If a sample with squares is used, the pitch in X and Y should be around 10  $\mu\text{m}$ . The height of the features in Z should be around 100 nm.

1. Scan a standard sample with known dimensions in X, Y and Z in both X ( $0^\circ$ ) and Y ( $90^\circ$ ). It is helpful to ensure that the sample is inserted with the features aligned at  $0^\circ$  and  $90^\circ$  before scanning.
2. Open the AFM Calibration software. Make sure to allow blocked content.



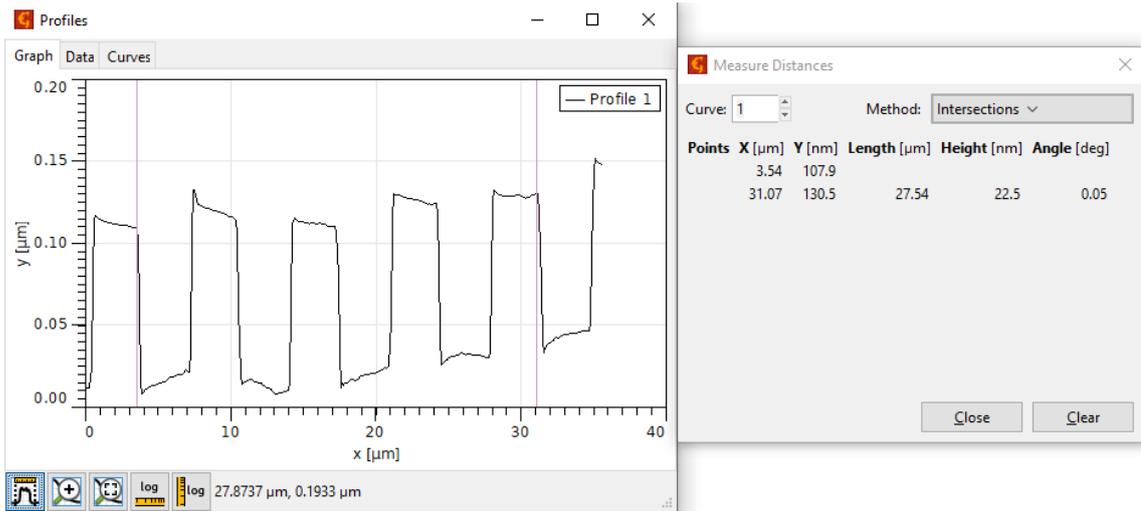
[Image C1: AFM Calibration software]

3. Insert the current calibration values, located on the System tab, under System Cal in the calibration software.
4. Insert the known values for the calibration standard in the Actual column in the calibration software.
5. To calibrate X, open the completed 0° scan in Gwyddion and level the image.
6. Extract a profile over a clean row of squares, in this case, horizontally, and click Apply.



[Image C2: Horizontal profile]

7. Using the Measure Distances feature on the bottom left-hand corner of the extracted profile, create vertical lines that bisect each square at approximately the same point. This is easiest using the corners of the features.



[Image C3: measuring profile]

8. Insert the values in the Length column into each X Measures blanks in the calibration software.

Welcome to the AFM Workshop calibration form. X, Y, Z, Z\_Sense  
 Don't forget to allow ActiveX Control on Launch if prompted!  
 Tested working with Google Chrome and Internet Explorer.

Piezo	System Cal	Measures1	Measures2	Measures3	Measures4	Actual
X	0.65	6.885	7.105	7.08	6.98	10
Y						
Z						
Z_Sense						
						Set XY

Calculate

Reset

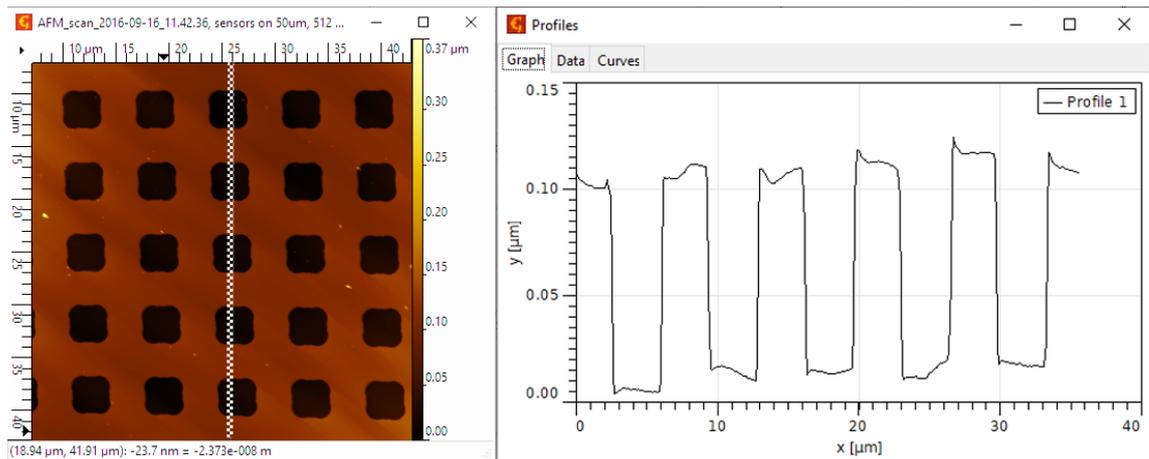
New X:  
 New Y:  
 New Z:  
 New Z\_Sense:

Update Calibrations

Clear X Clear Y Clear Z Clear Z\_Sense

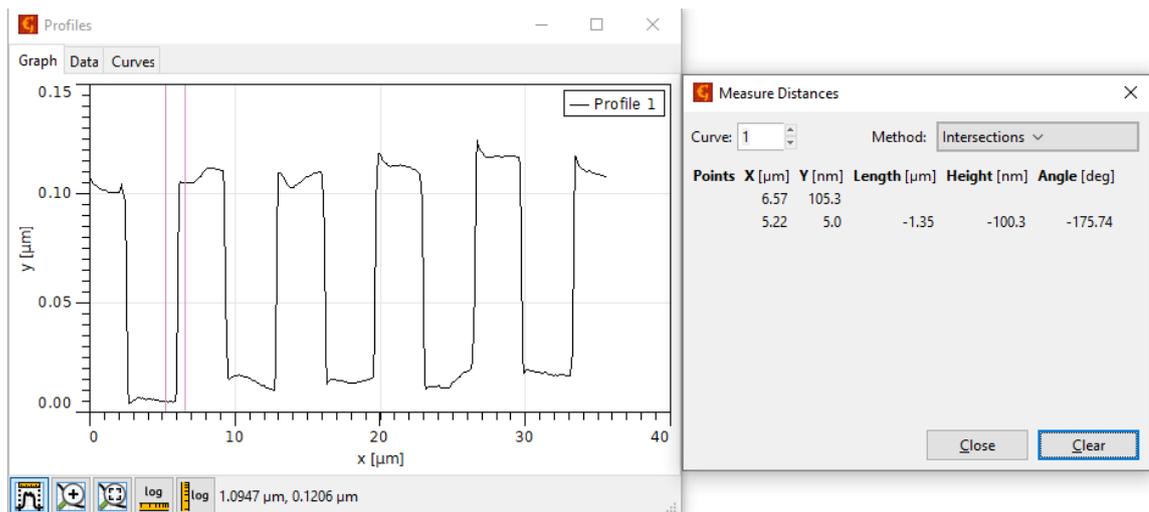
[Image C4: using applet]

9. Repeat steps 5 - 8 with the completed 90° scan to obtain Y Measures values. Make sure to extract a profile along the best looking column of squares in the Y-direction.



[Image C5: Y-profile screenshot]

10. To calibrate Z, the user may either use the 0° or 90° scan. Extract a profile, and measure distances from the top of each square to the bottom of each pit. Enter the Height values in the Z Measures blanks in the calibration software.



[Image C6: calibrating Z screenshot]

11. Once the calibration software is completely filled out, select Calculate to obtain the new calibration values. Enter these values in the System tab and click Save.

Welcome to the AFM Workshop calibration form. X, Y, Z, Z\_Sense  
 Don't forget to allow ActiveX Control on Launch if prompted!  
 Tested working with Google Chrome and Internet Explorer.

Piezo	System Cal	Measures1	Measures2	Measures3	Measures4	Actual
X	0.65	8.56	8.68	8.7	8.4	10
Y	0.65	9.1	8.95	9.19	9.05	10
Z	0.70	102	103	99	107	112
Z_Sense	2.2	91	89.5	93	89.7	112
						Set XY

Calculate

Reset

New X: 0.5428654630168901  
 New Y: 0.5835491871038853  
 New Z: 0.7630170316301702  
 New Z\_Sense: 2.713656387665199

Update Calibrations

Clear X   Clear Y   Clear Z   Clear Z\_Sense

[Image C7: filled in calibration applet form]

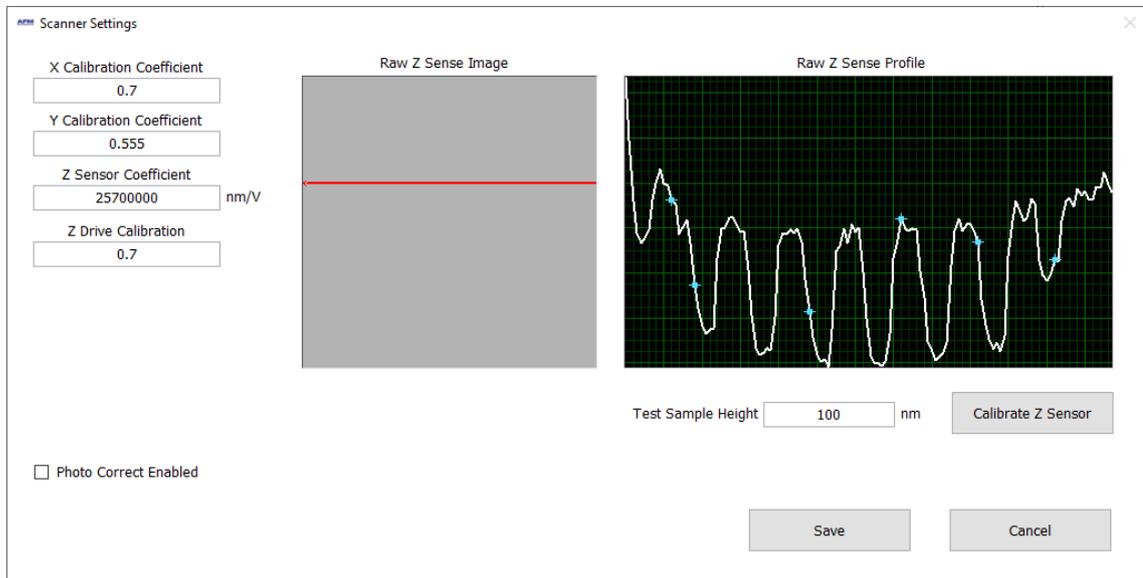


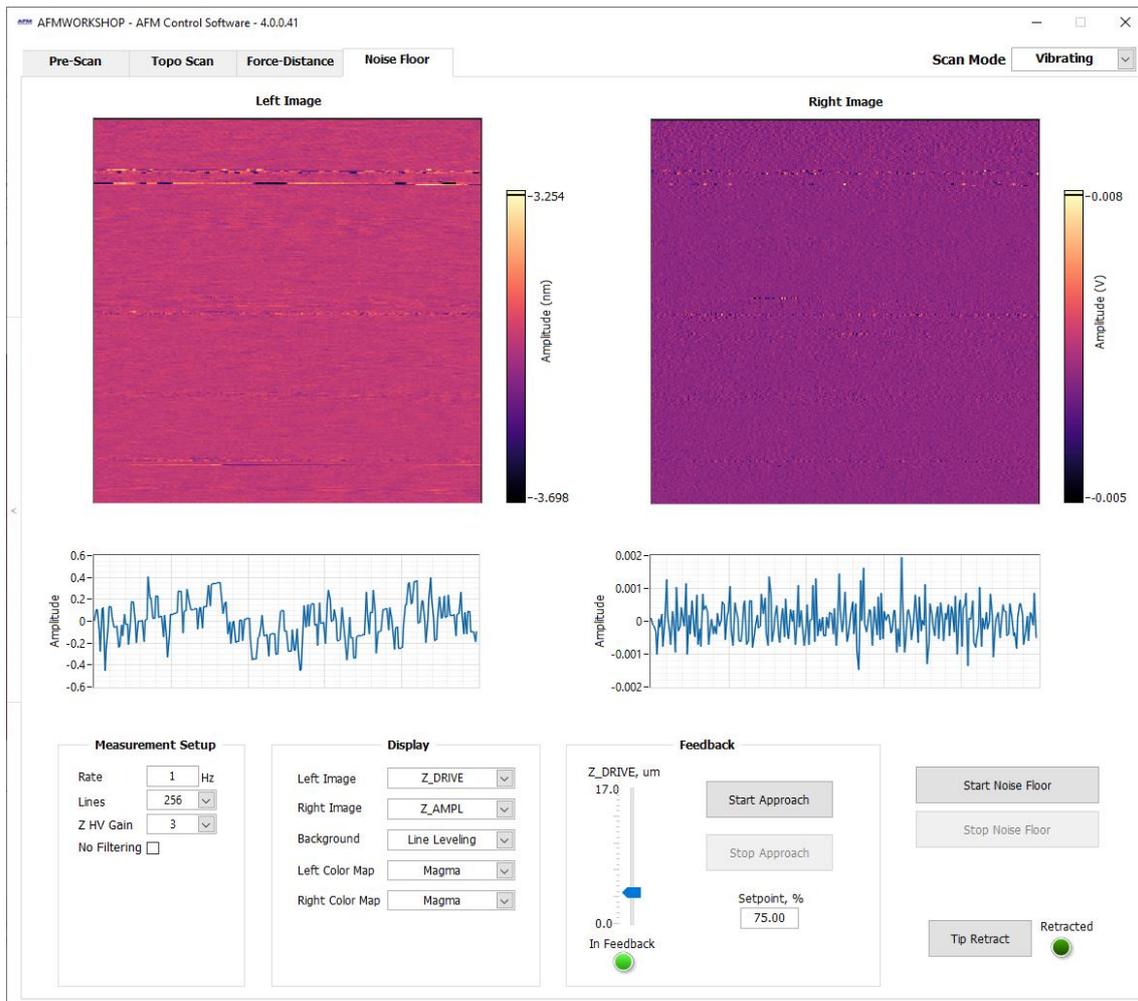
Image C8 entering values in to the scanner calibration form in software

**NOTE:** To obtain the most accurate calibration values for the scanner, the user may have to complete multiple calibration iterations

## Appendix D: Noise Floor

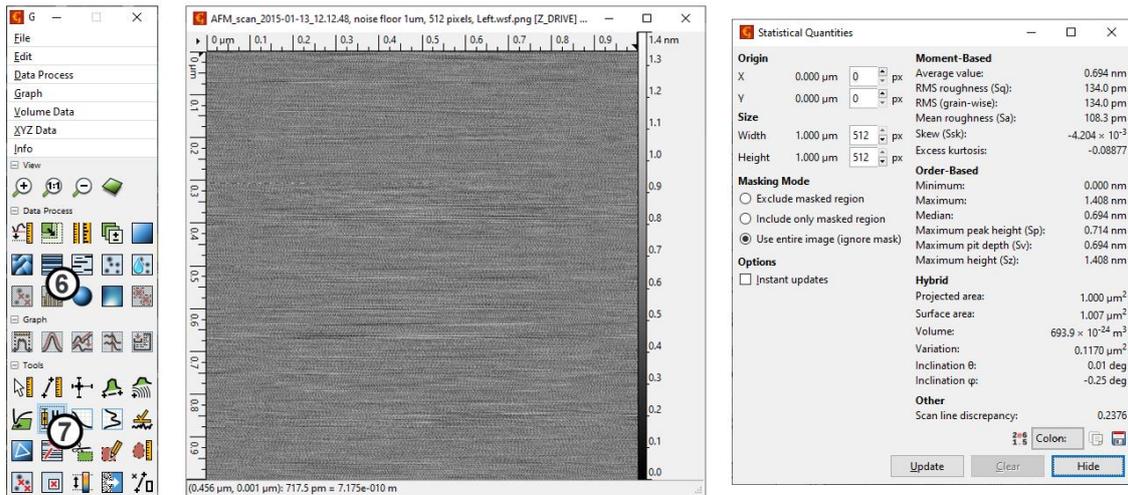
To measure the noise floor, it is necessary to scan the sample with no voltage applied to the X and Y piezoelectric ceramics. The AFM Control Software will then measure the structural noise of the system when the tip is in feedback with the surface. There is a special routine built into the AFM Control Software for this purpose. Make sure the tip is a sizeable distance from the surface. Initiate the Tip Retract function, if necessary.

1. Press the key combination CTRL-N in order to activate the Noise Floor measurement tab, as shown below



[Image D1: Noise floor tab]

2. In the noise floor tab, set the scan parameters for noise measurements:
  - Scan Rate = 1 Hz
  - Scan Lines = 256
  - Left, Right Image = Z-Drive, Z-Error
3. Initiate Automated Tip Approach.
4. Begin a scan.
5. To analyze the noise floor measurement, open the completed Z-Drive scan in Gwyddion.
6. Level the image by pressing the Polynomial level button, 1<sup>st</sup> order.
7. Find the noise value with the Statistical Quantities button, under Ra (Sa) for surface roughness.



[Image D2: Noise floor analysis]

Noise can be reduced by lowering the Z HV Gain using the Z HV Gain setting in the Noise floor tab. This should be done with the probe out of contact with the surface. When the Z gain is lowered, a new tip approach must be initiated because the Z ceramic's dynamic range has been reduced.



**CAUTION:** Increasing the Z HV Gain will increase the Z ceramic's dynamic range, and will raise the sample stage. Make sure the tip is removed from the surface before increasing Z HV Gain.

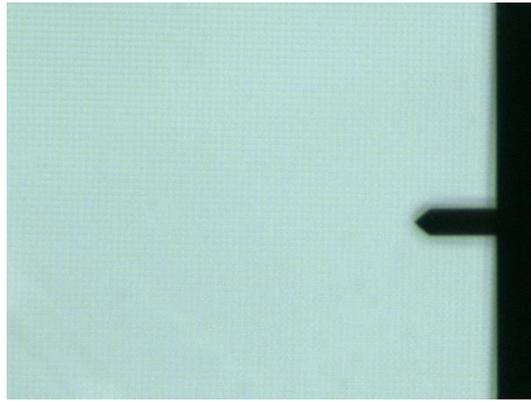
## Appendix E: Probes

There are two major types of probes, vibrating mode and non-vibrating mode probes. Each of these probes may have a metallic coating, which makes them more reflective.

### 1. Vibrating Mode Probes

Vibrating mode probes are in the shape of a rectangle. Typical VM probe specifications:

<b>Resonance Frequency</b>	150 – 350 kHz
<b>Length</b>	125 – 225 $\mu\text{m}$
<b>Width</b>	30 – 40 $\mu\text{m}$
<b>Thickness</b>	0.5 – 1 $\mu\text{m}$
<b>Force Constant</b>	15 – 50 N/m



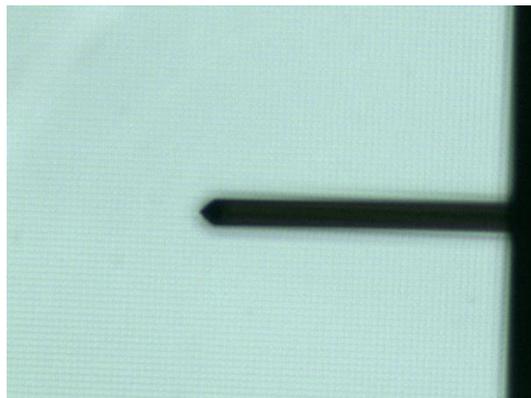
[Image E1: video microscope image showing view of ACLA]

ACLA probes from Applied Nanostructures are recommended.

## 2. Non-Vibrating mode (contact) Probes

Non-vibrating mode probes can be in the shape of a V or rectangular. Typical Non-Vibrating mode probe specifications are:

<b>Resonance Frequency</b>	7 – 200 kHz
<b>Length</b>	225 – 450 $\mu\text{m}$
<b>Width</b>	30 – 40 $\mu\text{m}$
<b>Thickness</b>	0.1 – 0.5 $\mu\text{m}$
<b>Force Constant</b>	0.1 – 0.5 N/m



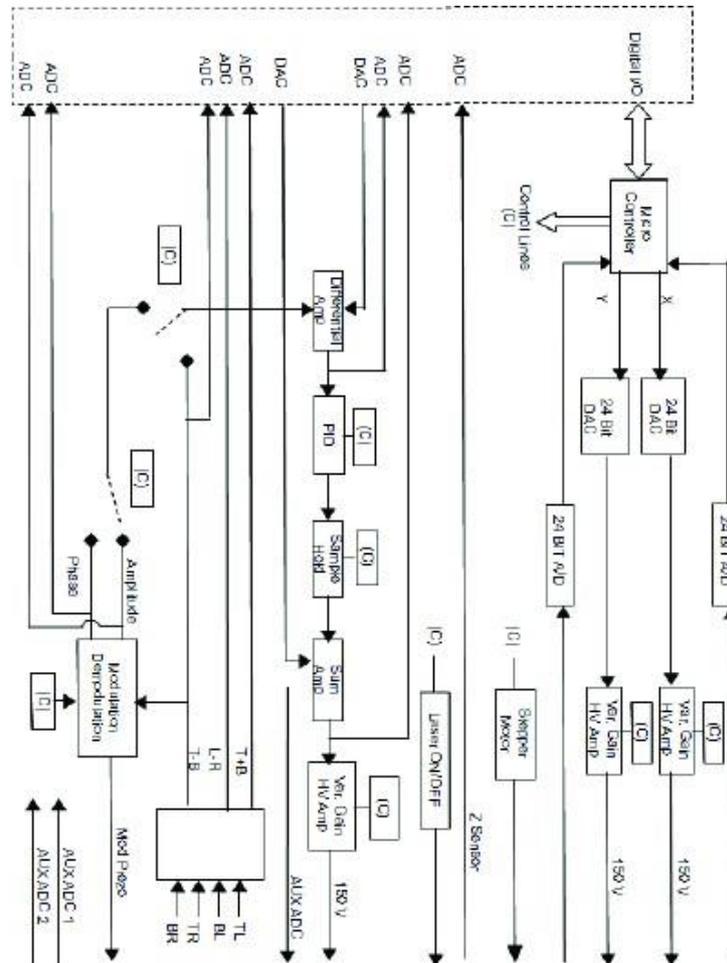
[image E2: video microscope image of SHOCONA]

SHOCONA probes from Applied Nanostructures are recommended.

## Appendix F: Technical Information

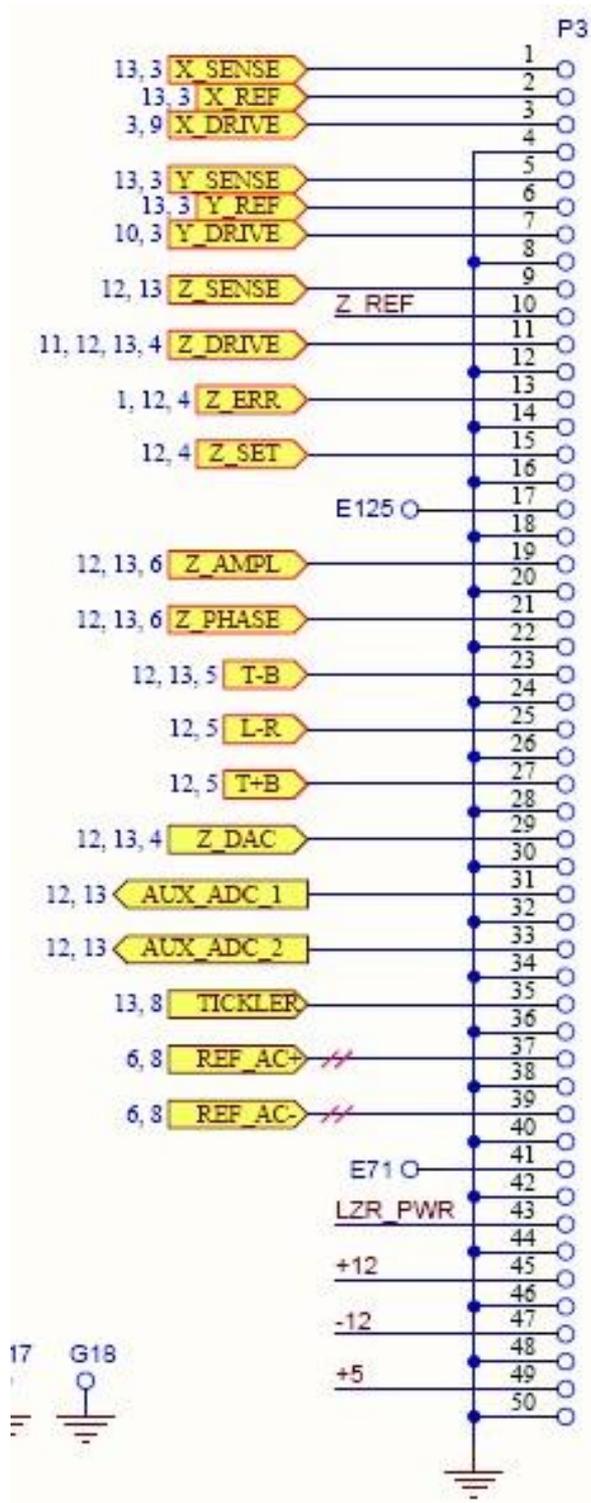
This appendix has technical information that may be useful for creating customer instrumentation with the HR-AFM. Additional information is available in the HR-AFM technical guide.

### F1 Electronic Block Diagrams



[Image F1: block diagram figure]

### F.2 Ebox and Modes Pin Assignment



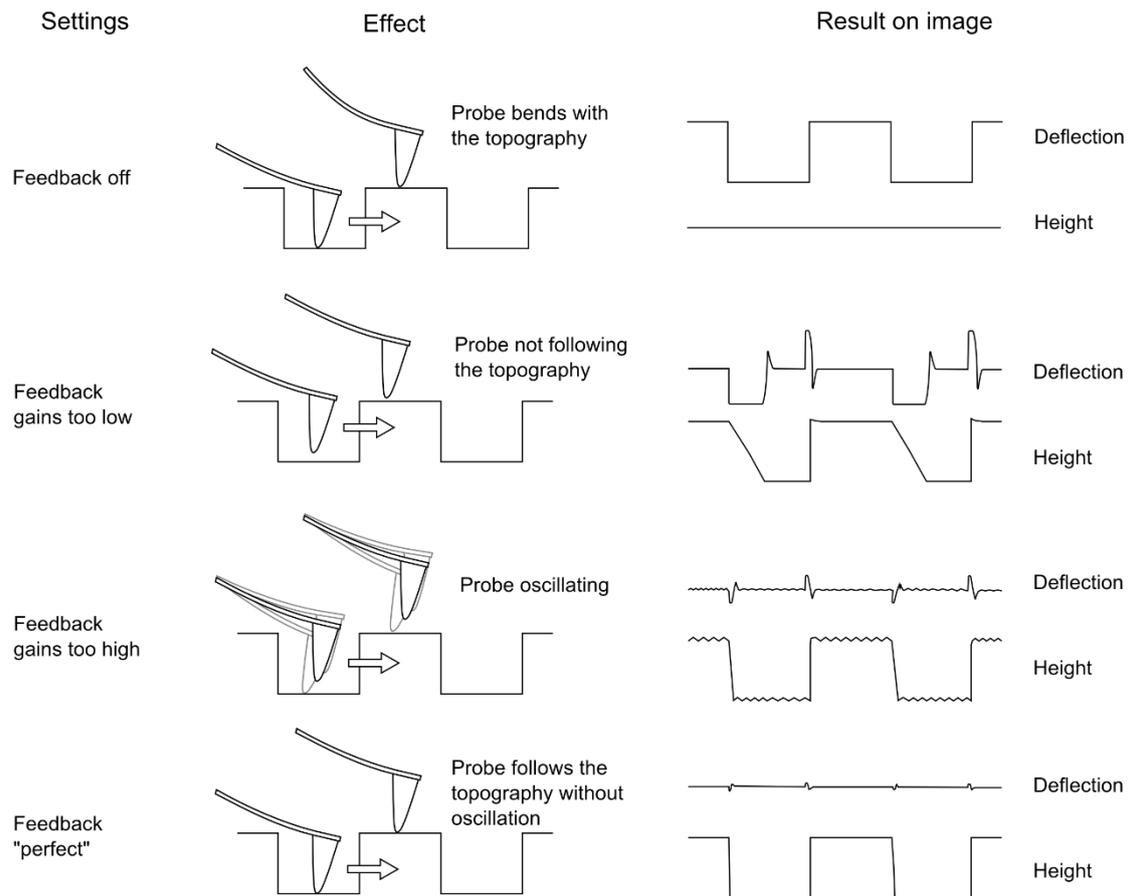
[imageF2: Cable connector figure]

### Appendix G: Optimizing GPID Parameters

The Z feedback scan parameters, such as the setpoint voltage and the GPID parameters are adjusted as the line scan is being made. The goal for adjusting these parameters is to have the probe perfectly track the surface. The probe is tracking the

surface best when the Z error image has a minimal signal. Establishing the optimal conditions requires practice and care.

When first learning to operate an AFM, it is helpful to scan a reference sample in 2D and adjust the GPID settings to see the effect on the Z voltage and the Z error signal, as shown in the figure below. The Option for 2D imaging is located on the topo scan tab, and will continuously scan the same image line over and over, allowing the user to easily see the effect of adjusting GPID and setpoint parameters. The image below schematically shows the effect of GPID parameter s that are too low or too high.

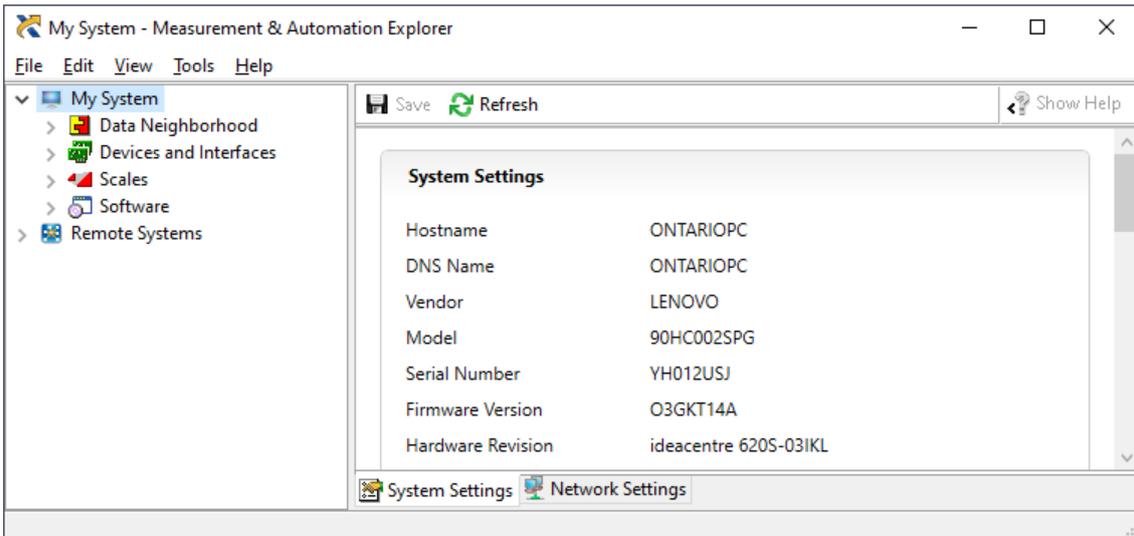


[Image G1: Feedback optimization]

## Appendix H: Renaming National Instruments DAQ Device

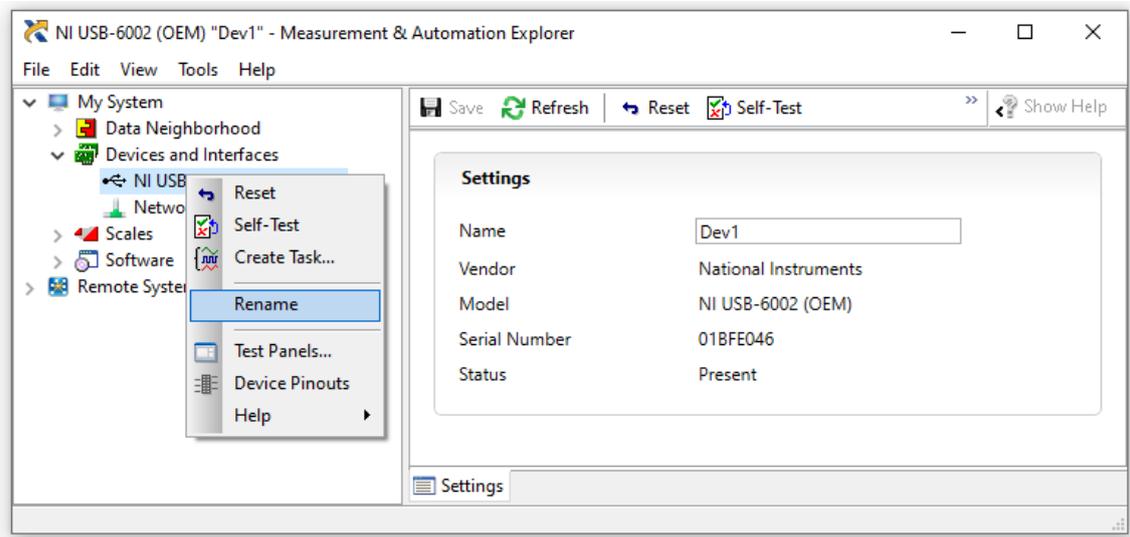
The following procedure will allow the user to reset the National Instruments DAQ card registry with the device number corresponding with the user's Ebox. This may be useful if the user's AFM Control Software does not initialize. This unusual occurrence can occur if multiple version of the software have been installed.

Open the NI MAX: Measurement and Automation Explorer software as follows:



[Image H1: screen shot with NI MAX]

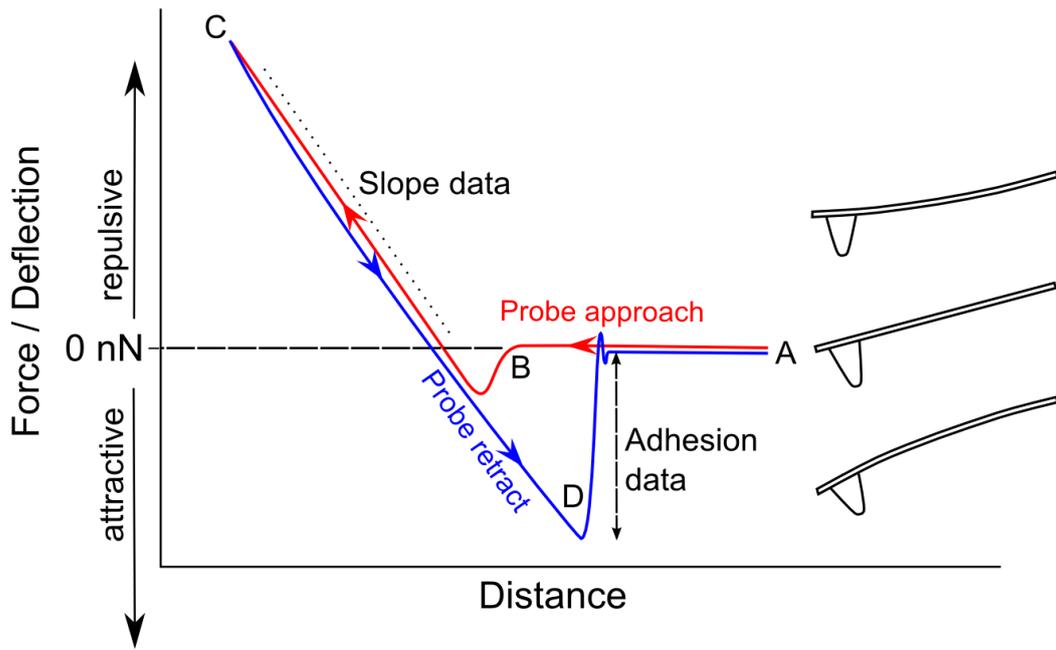
1. Expand Devices and Interfaces under My System on the left scroll tab.
2. Making sure the correct Ebox USB is plugged into the computer, delete any devices that are not green. There should only be one green device, corresponding with the DAQ card in the user's Ebox.
3. Rename the only remaining green device as Dev1. This can be accomplished by right-clicking on the device and selecting Rename.



[Image H2: renaming]

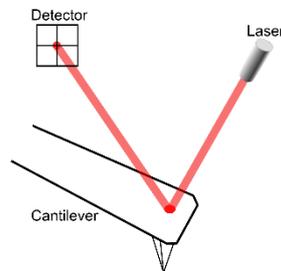
## Appendix I - Analyzing Force/Distance Curves

A force/distance curve is a graph that shows the amount a cantilever tip bends as it is systematically pushed into a sample. The probe may be lowered onto the surface, or the surface may be raised into the probe.



[Image I1: force distance curve]

What is actually measured is the deflection of the cantilever, or the Top - Bottom (T-B) signal of the photo-detector.



[Image I2: Light lever figure]

The deflection of the cantilever can be converted to Force - usually expressed In NanoNewtons (nN) - using Hooke's law:

$$\text{Force} = - K \times d$$

K = force constant (nN/nm)

d = deflection of the cantilever (nm)

In a light lever AFM, the user must convert the T-B signal into the cantilever deflection. This is achieved by measuring the change in the T-B signal as the sample is directly

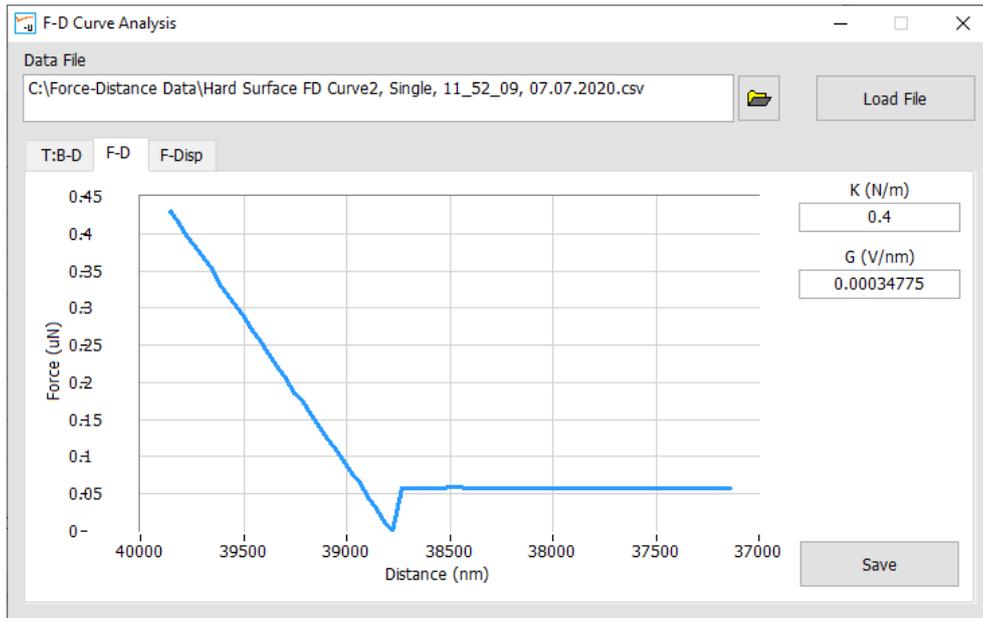
pushing against the probe. This is labeled slope data in the first figure above. The force constant for the cantilever is supplied by the manufacturer.

A force/distance curve from the HR-AFM is illustrated in the figure below. The light lever gain can be determined simply in any spreadsheet application. However, the AFMWorkshop F-D Curve Analysis applet was used below.



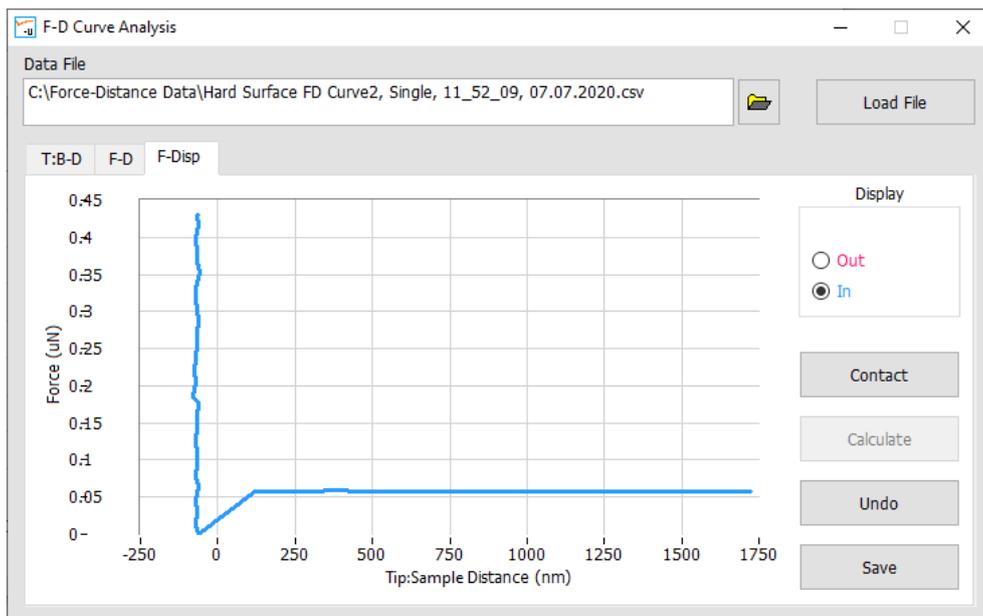
[Image I3 Deflection-distance curve]

The data above shows the Deflection vs. distance curve, which can be used to calculate the light lever gain. This was calculated using the region between the two black lines. As shown this was found to be 0.3 mV /nm. The second image below uses this value, along with the value of spring constant (K) to convert the data into a force-distance curve.



[Image I4 Force-distance curve]

The final image below shows the curve converted to Displacement-distance curve. In the case where no indentation or compression of the sample occurs, as shown here, the curve shows a vertical line upon contact with the surface. This indicates that the probe was not moving at this point, and the only change was bending of the probe. In experiments such as nanoindentation, there will be a slope in this region, However, a curve as shown here is typically used to calibrate the system during each experimental run.



[Image I5 Force-Displacement curve]